



August 16, 2001

Ms. Bonnie Lavelle
Work Assignment Manager
EPA Region VIII, 8EPR-SR
999 18th Street, Suite 500
Denver, CO 80202

REF: RAC No. 68-W7-0039
WA Nos. 004-RICO-089R, 007-RICO-085G, 007-RICO-085G

SUBJECT: WAF Revision, Request for Additional Information on Audit of Paragon Analytics, Inc.

Dear Bonnie:

In your subject Technical Direction Memorandum, you requested the following information:

1) Information on the Period of Concern

Washington Group International, Inc. (WGI) entered into an agreement with Paragon for services to the RAC on October 22, 1999. The award to Paragon was based on a competitive bid process and review of their written LQAP.

- a. WGI has performed one audit (see Enclosure 1) of Paragon Analytics, Inc. (Paragon). The VB/I-70 work plan states that a laboratory audit will be performed "if requested by the WAM." During an April 12, 2001 VB/I-70 status meeting, Marta Green recommended that we should proceed with the audit, considering that the post-RI sample results would be generated by Paragon (as opposed to the primary prior use of Paragon to perform quality control checks on the XRF results). We were then verbally directed to proceed with the audit.
- b. Paragon agreed to provide copies of all audits performed in support of Federal contracts. These reports and their responses are attached (Enclosure 2).

2) Completeness of the Audit Report

WGI apologizes for the mix-up in versions of the audit report sent to the EPA and the consternation this has caused. Revision 1 of the Paragon Audit Report (No. RAC-V-01-01) has been provided (see Enclosure 1). We have initiated training on the Washington Group document revision procedures for all RAC personnel to ensure that this does not happen again.

There were errors in the checklist attached to the report, which resulted in the unsatisfactory ratings without a corresponding Audit Finding Report or Audit Observation Report. Please note that the corrected composite audit checklist (Enclosure 1) has deleted items 44 (a duplicate of item 42) and 46 (a duplicate of item 41). The following list addresses each of these items and ties them to the enclosed corresponding objective evidence:

Audit Checklist Items:

3. Are MDLs run on each instrument and each matrix? This item is satisfactory ("Sat"). See attached representative MDLs provided by Paragon for review (Enclosure 2). Paragon analyzes MDLs on each instrument for each matrix and then chooses the worst case scenario to load into the LIMS system for reporting purposes.

4. Review and verify that MDLs are run at a frequency that provides consistency in meeting the Method Reporting Limit (RL). Are MDLs run annually? This item is satisfactory. See attached MDLs for the dates analyzed (Enclosure 2). Please note that none of the MDLs are expired.

11. Verify that calibration standards are chosen to bracket the expected concentration levels of the parameter contained within the sample. Ensure that calibration standards are prepared at a minimum of three concentration levels or (3-5 times) and (5-10 times) the estimated MDL plus a calibration blank. This item is satisfactory. See attached method specific calibration data from Paragon showing the concentrations and levels run (Enclosure 3).

12. Verify that the accuracy of prepared standards is periodically checked by comparison with a standard from an independent source. Additionally, verify that a second source standard or initial calibration verification (ICV) is run after the initial calibration and the responses are compared against one another. This item is satisfactory. See attached calibrations containing second source calibrations which were analyzed after the ICAL and compared against it (Enclosure 3).

16. Verify that when GC/MS is performed the following operational parameters are adhered to satisfy analytical requirements associated with the determination of organic compounds in water and soil sediment:

- **Documentation of GC/MS mass calibration**
- **Documentation of GC/MS response factor stability**
- **Internal standard response and retention time.**

This item is satisfactory. See attached calibrations (Enclosure 3) containing the Form 5 (showing BFB or DFTPP tunes), Form 6 and 7 (showing response factors and RSDs) and the calibration raw data (showing the internal standard areas and the retention times).

35. Verify the following licenses, accreditation and certifications are held and maintained as applicable to Washington Group's subcontract:

- **State of Colorado Department of Health**
- **State of Utah Department of Health**
- **State of California Department of Health Services**
- **State of Arizona Department of Health Services**

This item is satisfactory. None of the certifications were expired on the day of WGI's audit. The State of Utah certification was due to expire in May of 2001. See the new State of Utah certification attached (Enclosure 4), which has a date effective of May 31, 2001.

3) Information on the Scope of the Findings

The findings were all programmatic or global in scope except for the two expired SOPs (AFR No. 1). Paragon SOP 409 (PCBs) referenced EPA SW-846 Method 8082 and SOP 525 (GC/MS VOAs) referenced EPA SW-846 method 8260B. Please note that the attached expired SOPs (Enclosure 5) are referencing the current versions of the methods involved and just need to be revised in order to reflect any changes in laboratory practices since the last SOP revision. Paragon has also submitted copies of their most recent control charting for representative methods in response to WGI's request (AFR No. 3). See attached documents showing the results (including method, matrix, analyte and dates) used for determination of the laboratory's in-house control limits (Enclosure 6).

You also requested information about the data validation process. Each data package is subject to Paragon's internal quality review before it is released as a final data set. See attached Paragon Case Narratives (Enclosure 3) showing the names and dates for two levels of review before the data results are considered final. Additionally, a third level of review is done on select data packages by the project managers and QA staff.

Washington Group performs an independent validation of the analytical data packages. The Washington Group Project Chemist performs a "Level 3" validation on 100% of all data, and performs a "Level 4" validation on 10% of the data. The data validation is performed in accordance with Washington Group's RAC Technical Standard Operating Procedure for Data Validation (TSOP-3), which is based on the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (OSWER 9240.1-05A-P October 1999) and Inorganic Data Review (9240.1-05-01 February 1994). A copy of TSOP-3 was most recently submitted to EPA on July 17, 2001 as part of the RAC Phase II Investigation Sampling Analysis Plan for the Remedial Investigation at Intermountain Waste Oil Refinery (IWOR).

Please contact me if you have any questions or require additional information. We would be happy to meet with you, at your convenience, to discuss any remaining questions or outstanding issues related to this audit.

Sincerely,



Kathy Wolf
VB/I-70 Site Manager

Enclosures

1. RAC-V-01-01, Revision 1
2. External Audit Reports/Paragon Responses
3. Paragon MDLs
4. Paragon Calibration Data/Case Narratives
5. State of Utah Certification
6. Paragon SOP 409 and SOP 525
7. Paragon Control Charts

cc w/encl.:

T. Medrano, 8TMS-Q

cc w/o encl.:

M. Goldade, 8EPR-SR

A. Hamp, 8TMS-G

L. Lloyd, 8EPR-SR

J. Powell, 8EPR-SR

P. Bell, Washington Group

M. Green, Washington Group

D. Lambert, Washington Group

A. Sacha, Washington Group

ENCLOSURE 1:

RAC-V-01-01, Revision 1



August 16, 2001

Ms. Debra Henderer
Quality Assurance Manager
Paragon Analytics, Inc.
225 Commerce Drive
Fort Collins, CO 80525

SUBJECT: Washington Group International, Inc. "Revised Quality Assurance Audit Report of Paragon Analytics, Inc."

Dear Ms. Henderer:

Enclosed for your review and response is Washington Group International Inc. revised Quality Assurance Audit Report No RAC-V-01-01, Revision 1 of audit activities at Paragon Analytics, Inc. located in Fort Collins, Colorado. The audit was conducted on May 08, 2001 to verify, by examination and evaluation of objective evidence, the ability of your laboratory to provide Chemical Analytical Analysis. In addition, the scope of this audit was inclusive of verifying Paragon Analytics' Inc. capability to perform work as stipulated in the October 18, 1999 Subcontract 1D9-4994-SC01.

Pursuant to the initial audit report that was transmitted to you and our conversation on August 15, 2001 we are transmitting to you our revised Audit Report. The audit report reflects some minor changes that were incorporated as a result of omissions and clarification following EPA's review and comment of the report. Please note that an additional observation, as well as our revised checklist, which corrects items that were incorrectly identified as unsatisfactory, were included in the revised report. Observation No.3 was issued as clarification and correlates to item number 10 contained within the audit summary. This item is included as a noteworthy item and requires no response.

The audit resulted in seven (7) Quality Findings and three (3) Observations, which are documented in the attached report. The revised report should not impact the corrective actions and response that you are currently initiating. However, it should be re-emphasized that the following items must be addressed for each Audit Finding Report and Observation No. 1:

- a. The steps, which have or will be taken to correct the condition reported;
- b. The root cause that led to the condition reported;
- c. The steps taken to prevent recurrence;
- d. Lessons learned (if applicable);
- e. The dates when indicated action was or will be completed.



Corrective Actions to all items requiring response shall be both concise and to the point. All items requiring response will require verification of corrective action implementation and re-evaluation.

The revised audit report is attached for distribution to the appropriate personnel or department heads for inclusion of the required responses. Please submit your responses in the spaces provided on the attached revised report. The original report should then be transmitted back to the Denver Regional Quality Assurance office for evaluation.

Should you have any questions regarding our approved vendor program or this revised report please contact me at (303) 843-3204

Sincerely,

Paul M. Bell

A handwritten signature in cursive script that reads 'Paul M. Bell'.

Washington Group International

PMB



Washington

**WASHINGTON GROUP INTERNATIONAL, INC. QUALITY ASSURANCE
AUDIT REPORT NO. RAC-V-01-01**

Date 05/08/01

TO: Ms. Debra Henderer

FROM: David C. Lambert

LEAD AUDITOR:

(Signature)

Paul M. Bell for David C. Lambert
AUDIT DATES: May 08, 2001

RESPONSE DUE DATE: July 8, 2001

ORGANIZATION Washington Group International, Inc. (Denver Regional Office)

ACTIVITY AUDITED: Paragon Analytics, Inc. Laboratory Quality Assurance Activities

PURPOSE/SCOPE: The scope of this audit was to evaluate Paragon Analytics Inc. implementation of laboratory quality program for activities and environmental testing protocols being performed at their facility in Fort Collins, CO. This audit was performed in support of the U.S. EPA Response Action Contract (RAC). These projects are inclusive of the Vasquez Boulevard/Interstate-70 (VB/I-70) site in Colorado, the Intermountain Waste Oil Refinery (IWOR) and the Eureka Mills site in Utah. The audit was initiated to verify compliance with Quality Assurance guidelines specified in both the VB/I-70 Phase IIIB QAPP and the IWOR Phase I QAPP.

AUDIT TEAM:

- Team Leader – D. C. Lambert
- Auditor – P. M. Bell
- Subject Matter Expert – A. Sacha

PERSONNEL CONTACTED DURING AUDIT:

Name

Title

See Attachments A and B

SUMMARY:

The purpose and scope of the audit was presented at a pre-audit conference held on May 08, 2001, at Paragon Analytics facility located in Fort Collins, CO. The audit was performed in accordance with a written checklist of applicable laboratory QA program requirements. The audit results were derived based on interviews of personnel, review of records and logbooks, inspection of instruments, and the evaluation of QA Program procedure implementation. Audit results were presented to the appropriate Paragon Analytics, Inc personnel at the post-audit conference held on May 08, 2001.

The audit resulted in seven (7) minor Findings and two (2) Observations which are included in the attached report. With the exception of the noted Findings and Observations, the audited Laboratory QA

Program elements and criteria were determined to be in compliance with the QA program requirements and effectively implemented.

The audit team would like to thank all Paragon personnel contacted during the course of this audit.

The following good laboratory practices or noteworthy items were observed during the audit investigation and all responsible personnel should be commended for their professionalism.

- All employees are provided with dosimeter badges to be worn while in radiation areas.
- General laboratory housekeeping was good throughout the laboratory.
- Current staffing levels and evening/weekend coverage are excellent in regards to urgent turn-around times.
- Internal Chain-of Custody forms used for sample receipt to analysis to archival to disposal are organized and fully implemented.
- Good chemical hygiene was observed by the use of MSDS sheets, clear labeling of chemicals, solvents and standards. Containers in use were noted to retain the appropriate custody log-out documentation inclusive of the analyst initials and opened date on the container.
- The waste generation and disposal program currently in place is outstanding.
- All customer service provided to date by the Project Manager has been excellent.

In conclusion, the Paragon Analytic's Laboratory sample analysis and data validation is within acceptable limits to meet Washington Group International Inc. needs, provided the deficient items addressed throughout this audit report are satisfactorily corrected and verified through follow-up.

1. ORGANIZATION AND RESPONSIBILITIES

The organizational structure is adequately described in the Paragon Analytics LQAP, Section 2, and further illustrated in Appendix A of the LQAP. Activities and responsibilities are further defined and delineated in the LQAP.

Satisfactory compliance.

2. LABORATORY QUALITY ASSURANCE PROGRAM

The Paragon Analytics' Laboratory Quality Assurance Plan (LQAP Revision 4, dated 02/99) was reviewed. The frequency of internal reviews and revisions to the LQAP as stated are not being performed within the established frequency of once every two years. Review of Paragon Analytics LQAP indicated that many stated procedural requirements are not currently being practiced in the laboratory. A review of LQAP Section 16.2 revealed that Paragon was previously classified as a small quantity waste generator whereas now, Paragon is classified as a large quantity waste generator. Further review of Paragon LQAP, Section 15.1, stated that all laboratory employees who engage in laboratory activities are required to submit to annual physical examinations in accordance with the Laboratory's Medical Surveillance Program.

Nine (9) Laboratory Standard Operating Procedures (SOPs) were reviewed. SOP 409, Revision 0, (PCB Analysis), and SOP 525, Revision 4, (GC/MS VOA Analysis) were not up-dated bi-annually as specified in Paragon LQAP Section 1.5.2.

Additionally laboratory control limits and the associated control charts were reviewed. However, laboratory control limits and the control limit update frequency were not being re-calculated annually or semi-annually as required by US EPA Method SW-846-8000B, Section 8.7.5. During this audit, there were no records or personnel files to substantiate whether these programmatic elements are currently being implemented.

Reference Audit Finding Report (AFR) No. 01

3. QUALITY ASSURANCE OBJECTIVES

The objectives specified and defined within the Paragon Analytic's Laboratory Quality Assurance Program, Standard Operating Procedures and Program Specifications were reviewed during this audit. Review of various quality-affecting documents indicated that laboratory quality assurance objectives are being met through controlled distribution, preparation, and completion of laboratory protocols, with the exception of items identified throughout this report.

The majority of the laboratory activities were in compliance with laboratory procedures, with the exception of documents such as; (LQAP annual review, control limit calculations, and training records) which do not currently meet the objectives outline in Revision 4 of the LQAP.

Reference Audit Finding Report (AFR) No. 02

4. SAMPLE PRESERVATION, HOLDING TIMES AND HANDLING PROCEDURES

Sample preservation, holding times and handling procedures were reviewed. The laboratory sampling, preservation and handling protocols were assessed to ensure that scientific data is legally defensible and are in accordance with the protocols specified by USEPA Contract Laboratory Program.

Satisfactory compliance.

5. SAMPLE CUSTODY

Sample Internal Chain-of-Custody compliance was verified by visual inspection of the Sample Custody receipt and storage area. All sample custody activities inclusive of chain-of-custody, data validity, checkout and storage were verified as meeting the appropriate U.S. EPA requirements.

Satisfactory compliance.

**6. ANALYTICAL PROCEDURES**

Analytical Procedures were reviewed to verify compliance to the analytical protocols prescribed by various EPA Methods and compliance to the detailed requirements specified in each respective procedure. During the course of the audit, the audit team noted observations regarding analytical procedural protocols as follows:

- There is currently no solvent testing program in place (as specified by LQAP Section 17.2)
- Monthly supervisory reviews of laboratory logbooks are not being performed on a routine basis

Unsatisfactory Compliance

Reference Audit Observation Report (AOR) No.1

7. CALIBRATION PROCEDURES AND FREQUENCY

Calibration procedures and calibration frequencies were reviewed. The requirements for the calibration of laboratory scales/balances, and the calibration of instrumentation used throughout the laboratory was verified and validated against instrument calibration logs. Calibration frequencies are being maintained as well as, calibration stickers were verified as being affixed to instruments that required calibration.

Satisfactory Compliance

8. PREVENTIVE MAINTENANCE

The Paragon Preventative Maintenance Program was reviewed for adequacy and effectiveness. During the audit, a broken and/or not in use GC/MS pump and GC OI Purge and Trap was observed in an auspicious location. Further investigation indicated that the GC/MS pump and GC OI Purge and Trap were not labeled with the appropriate status indicator or tag-out tag as specified by SOP 319.

Unsatisfactory

Reference Audit Finding Report (AFR) No. 4

9. QUALITY CONTROL PROCEDURES

Internal Paragon Laboratory Quality Control Procedures were reviewed to determine the in-house systematic process controls implemented to measure and detect errors or out-of-control events. In-house quality controls are defined and implemented through various procedures. The criterion that is used to measure and analyze environmental data includes measurements of accuracy and precision. However, the control limit measurements that are required to reflect the degree to which the measured value approximates the actual or true value for a given parameter

and the control limits which influence bias in measurements are not being updated semi-annually or annually for some methods as required by EPA Method Protocols.

Unsatisfactory

Reference Audit Finding Report (AFR) No. 3

10. DATA REDUCTION, VALIDATION AND REPORTING

Data reduction, validation and reporting of information throughout the laboratory was reviewed and verified. Work Order No. 0103075 was reviewed. During the audit team's review the following observations were noted:

- Many organic laboratories were missing the annotation of the amounts of various standards added to samples during prep or analysis on the run log books
- Corrections to sample extraction and preparation laboratory worksheets for ignitability or GC pesticide data were not corrected with a single line through and initial and date.
- Manual integration was not being documented properly by analysts. A review of GC/MS SVOA and Pesticide data indicated that the "before and after" reason for integration and subsequent initial and date are missing.
- Case narratives are incomplete. A review of GC/MS SVOA narrative revealed that dilutions were initiated for WGI samples. However, the case narrative did not provide an explanation or reason as to why the dilutions were necessary, and an explanation ascertaining why undiluted samples did not have target compounds over the linear range.

UN-Satisfactory

Reference Audit Observation Report (AOR) No.1

11. PERFORMANCE AND SYSTEMS AUDITS

This verification included the review of performance and system audit schedules and completed audits.

The laboratory initiates two types of audits used to verify and assess laboratory compliance. A review of Paragon's audit program indicated that laboratory audits are being performed. However, internal performance and systems audits are not being performed at the frequency of once per month as specified in the LQAP.

Unsatisfactory compliance.

Reference Audit Finding Report (AFR) No.6

12. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Reports to management were verified by review of nonconformance reports. The audit team reviewed various nonconformance reports and corresponding dispositions. Routine NCR dispositions such as; "use-as-is", "reject" and/or "repair" are not marked on the NCR form. Objective evidence of the disposition process indicated that in many cases the disposition was recorded as "Document in a Narrative". Further investigation indicated that in most cases, the narrative is undefined and is not attached or part of the disposition and closure of the NCR.

The NCR system does not provide adequate confidence that the nonconformance reporting and subsequent corrective actions are being dispositioned to preclude recurrence and are being tracked from initiation through closure.

Unsatisfactory

Reference Audit Finding Report (AFR) No.5

13. CORRECTIVE, ACTIONS

Laboratory Corrective Actions were reviewed. The corrective action program is in place. However, a review of audit results and subsequent corrective actions indicate that follow-up of corrective action implementation strategies are not being initiated within two weeks of report issuance as procedurally required. A review of the audit log indicated that a series of audits were performed in 1999 and 2000. The corrective actions to these audits were not noted as being either closed or that the corrective actions were completed.

Unsatisfactory

14. PERSONNEL TRAINING

Washington Group International was provided Paragon Training Documentation records for review. There was no objective evidence to substantiate department/laboratory specific training or subsequent checklists. Review of training records indicated that there was missing documentation attesting to the analytical staff's credentials (i.e., resumes, educational backgrounds, diploma's etc.) Additionally the following training records were noted as being incomplete: required Paragon LQAP training, Radiation Training RCRA Training etc. The training documentation that was reviewed did not summarize each analyst initial proficiency demonstrations (as specified in SW-846 and Paragon LQAP, Revision4 Section 14.2.2.2)

Unsatisfactory

Reference Audit Finding Report (AFR) No. 02

15. LABORATORY SAFETY

The Paragon Laboratory Safety protocols were reviewed by both visual inspection of laboratory areas and of in place programs. In general, the laboratory safety programs and personnel exhibit adequate knowledge to safely perform their assigned duties. Health and safety training was reviewed for various laboratory personnel. The Paragon medical surveillance program, which is inclusive of an annual physical examination for all employees, engaged in laboratory activities, is required by procedure. Training records indicate that no Paragon personnel have been given an annual physical as specified in the LQAP.

Unsatisfactory

Reference Audit Finding Reports (AFR) No. 01 and No. 2

16. LABORATORY WASTE DISPOSAL

The laboratory waste disposal was reviewed for various waste streams. The waste streams that are being generated are now of significant enough quantities to classify the laboratory as a large quantity waste generator. Currently the LQAP Section 16.2 classifies Paragon Laboratory as a small quantity waste generator, which does not coincide with the current waste generator classification.

Unsatisfactory

Reference Audit Finding Report (AFR) No. 01

17. PROCUREMENT CONTROL

Various procurement records were reviewed to assure legibility, traceability to associated items and, that they accurately reflect the work accomplished. Procurement records indicate that secondary source standards are being purchased from a different supplier than primary standards. Additionally, some procurement documents are not being reviewed or approved by cognizant supervision for quality affecting requirements such as, Certificates of Calibration, certificates of purity, NIST traceability etc.

Unsatisfactory Compliance.

Reference Audit Observation Report (AOR) No. 01



COMPOSITE AUDIT CHECKLIST

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QUALITY ASSURANCE

Organization: Paragon Analytics, Inc.	Location: Fort Collins, CO	Evaluation Date(s) 05/08/01
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Subject
Evaluation of Paragon Analytics, Inc. Laboratory Quality Assurance Program

References:
Paragon Analytics Laboratory Quality Assurance Program Revision 4 dated 02/99

Evaluation Performed by:

Dave C. Lambert Lead Auditor
Paul M. Bell Auditor
April Sacha Subject Matter Expert

Item	Attributes	References Paragon LQAP	Sat	Un-sat	N/A	Comments
1.	Verify that the latest revision(s) of SOP's are available and present in all laboratories. Additionally, verify that the following personnel have signed-off on the completed document: <ul style="list-style-type: none">Group Leader or technically competent staff memberLaboratory QA ManagerLaboratory Manager	Section 1.5.2	Sat			
2.	Verify that SOPs are distributed as controlled documents and QA has maintained a distribution list of each SOP.	Section 1.5.2	Sat			
3.	Are MDLs run on each instrument and each matrix?	Section 3.7	Sat			
4.	Review and verify that Method Detection Limits (MDLs) are run at a frequency that provides consistency in meeting the Method Reporting Limit (RL). Are MDLs run annually?	Section 3.7	Sat			
5.	Review internal chain-of-custody procedural protocols from receipt to archival. Are samples signed-out when removed for analysis? Ensure that the sample custody log references the following: <ul style="list-style-type: none">Sample identificationDate/timeAnalystLaboratory of analysis	Section 5.8.1	Sat			



COMPOSITE AUDIT CHECKLIST

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QUALITY ASSURANCE

Organization:		Location:			Evaluation Date(s)	
Item	Attributes	References Paragon LQAP	Sat	Un-sat	N/A	Comments
6.	Review and verify that all instruments used throughout the laboratory are traceable to NIST, EPA or other nationally recognized standards. Review and compare Paragon Equipment Lists of all major instrumentation. Sample equipment listed on the equipment list and the associated calibration certificates.	Section 7	Sat			
7.	Are all standards traceable? Review Standards Notebooks ensure that standards are stored in a manner as prescribed in Paragon LQAP Table 7-1.	Section 7	Sat			
8.	Verify that each standard is identified with an internal identification number. Ensure that stock standards are documented in the Standards Notebook by listing the following: <ul style="list-style-type: none">• Date of preparation• The analyst• The source of the reference material• Amounts used• Final volume• Serial number	Section 7.2	Sat			
9.	What is the GC/MS VOA preparation frequency for standards containing gases? Verify that the preparation frequency is documented. Review actual samples of gaseous standards.	Section 7.2	Sat			
10.	Are diluted working standards not consumed during an analytical session fully labeled, including the serial reference number of stock standards used in its preparation?	Section 7.2	Sat			
11.	Verify that calibration standards are chosen to bracket the expected concentration level of those concentration levels of the parameter contained within the sample. Ensure that calibration standards are prepared at a minimum of three concentration levels or (3-5 times) and (5-10 times) the estimated method detection limit plus a calibration blank.	Section 7.3	Sat			

COMPOSITE AUDIT CHECKLIST

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QUALITY ASSURANCE

Organization:		Location:				Evaluation Date(s)
Item	Attributes	Paragon LQAP	Sat	Un-sat	N/A	Comments
12.	Verify that the accuracy of prepared standards is periodically checked by comparison with a standard from an independent source. Additionally, verify that a second source standard is run after the initial calibration and the responses of the second source calibration and the standards are compared against one another.	Section 7.3	Sat			
13.	Verify that pH meters, balances and turbidity meters are calibrated daily with NIST traceable reference material. In addition ensure the following calibration frequencies are maintained: <ul style="list-style-type: none"> Analytical Balances – every 12 months entire range) Electrometer/pH – prior to use and once every four hours of use (calibrated with three buffer solutions) 	Section 7.3	Sat			
14.	Verify that Gas Chromatography user range calibrations are initiated by obtaining a three or five point calibration curve, consisting of all compounds of interest plus a calibration blank.	Section 7.6.1	Sat			
15.	Verify that the laboratory participates in the EPA-LV/EMSL Interlaboratory Comparison Program.	Section 9.2.2	Sat			
16.	Verify that when Gas Chromatography and Mass Spectrometry is performed the following operational parameters are adhered to satisfy analytical requirements associated with the determination of organic compounds in water and soil sediment: <ul style="list-style-type: none"> Documentation of GC/MS mass calibration Documentation of GC/MS response factor stability Internal standard response and retention time 	Section 7.6.2	Sat			
17.	Verify that water utilized to prepare most LCSs analysis is analyzed for conductivity and water dispensing stations are tested on a weekly basis and results are recorded on the Water Conductance Log sheets	Section 9.2.2	Sat			



COMPOSITE AUDIT CHECKLIST

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QUALITY ASSURANCE

Organization:		Location:			Evaluation Date(s)	
Item	Attributes	References Paragon LQAP	Sat	Un-sat	N/A	Comments
18.	Verify that the laboratory possesses a valid radioactive materials license.	Section 9.2.2	Sat			
19.	Are efficiency control charts plotted on a daily basis, reviewed by the QA/QC department once tri-monthly, when either graph value to be reported falls on or above the +2 sigma or on or below the -2 sigma is the QA department notified?	Section 9.4.2	Sat			
20.	Review and verify that for Method 8000B per section 8.7.5 control limits are update semi-annually. Additionally, review and verify the frequency in which laboratory control charts are updated.	Section 9.4.1		Un-Sat		Reference AFR No.3
21.	During Matrix Spike Sample Analysis, at what concentration percentage is each analyte in order to be within the linear range of the spiked sample solution. In addition is the acceptability of the control limit for a spike between 75-125% recovery.	Section 9.2.2	Sat			
22.	Verify that analytical spike sample analysis is being added after samples are prepared and prior to analysis and are run at a frequency of 5%.	Section 9.2.2	Sat			
23.	Verify that Laboratory Control Samples are ran independently with every batch of analysis and utilized for the verification of the internal standard from which the calculations are made.	Section 9.2.2	Sat			
24.	Verify that two (2) standard deviations are used for 95% confidence intervals during the calculation of control charts for the ICAP, and for each batch of samples analyzed the following QC checks are initiated: <ul style="list-style-type: none">• At least one blank analyzed• At least one LCS (spiked with all reported analytes• MS/MSD pair analyzed• One sample duplicate analyzed• One sample dilution (dilution factor =5)• Initial multi-point calibration (3 to 6 standards plus a calibration blank)• One-point calibration verification standard compared against the initial calibration curve• Second source calibration verification standard.• An interference check standard at the beginning and end of the run• Drift check standard analyzed between every 10 field samples and at end of analysis run	Section 10.2	Sat			

COMPOSITE AUDIT CHECKLIST

QUALITY ASSURANCE

Organization:		Location:				Evaluation Date(s)
	Attributes	References Paragon LQAP	Sat	Un-sat	N/A	Comments
25.	<p>Verify that tracking of standards, chemicals, and reagents used in inorganic chemistry are logged in a bound logbook and the following information is maintained within:</p> <ul style="list-style-type: none"> a. Date chemical/regent is opened b. Standard number c. Consecutive numbered tape d. Identification e. Manufacturer, lot number etc. d. Mixing information e. Noted mixing instructions f. Expiration date g. Shelf life instructions f. Numbering system 	Section 10.3	Sat			
26.	<p>Ensure that Level 2 reviews of data packages include the following:</p> <ul style="list-style-type: none"> • Group leader independent review • Calibration data are scientifically sound, appropriate to the method and completely documented. • QC Samples are within established guidelines • Quantitative identification of sample components is correct • Quantitative results are correct • Documentation is complete • Data package is complete. 	Section 10.3		Un-Sat		Reference AOR No. 3
27.	Review and verify that data reduction, validation and reporting are entered into the LIMS.		Sat			
28.	<p>Review and verify Paragon laboratory safety protocols. Are safety showers, fire extinguishers, etc., inspected? Additionally, verify the following:</p> <ul style="list-style-type: none"> • Hazard Communication Program including MSDS use. • Use disposal of chemical reagents, chemical standards, and analysis samples • Medical surveillance program including physical examinations of employees 			Un-Sat		Reference AFR No.1
29.	Is a record of Preventative Maintenance kept in the instrument log book for each piece of analytical equipment and is the task performed, date, and the person(s) performing the PM task logged into the log book?		Sat			



COMPOSITE AUDIT CHECKLIST

QUALITY ASSURANCE

Organization:		Location:				Evaluation Date(s)
	Attributes	References Paragon LQAP	Sat	Un-sat	N/A	Comments
30.	Verify that Level 2 reviews are structured to include 10 percent checks of calibration data and QC sample results and the results are against bench sheets. Additionally, when discrepancies Level 2 data packages are found, verify that an additional 10 percent of the samples are checked against bench sheets.	Section 10.4	Sat			Reference AOR No. 2
31.	Verify that the following internal audits are performed to assess and document performance of the laboratory staff in the following frequencies: a. Monthly Systems Audits	Section 11.1		Un-Sat		Reference AFR No. 6
32.	Review and verify that performance audits are documented and include the following: <ul style="list-style-type: none"> • Documentation of refrigerator blanks • Inspection/surveillance of temperature logbooks for refrigerators and ovens • Calibrations of mechanical pipettes 	Section 11.1		Un-Sat		Reference AFR No. 6
33.	Are audit results and subsequent corrective actions (e.g., follow-up) verified within two weeks of report issuance?	Section 11.1		Un-Sat		Reference AFR No. 7
34.	Review and verify the latest external systems audit of the following agencies: <ul style="list-style-type: none"> • State of Colorado Department of Health • State of Utah Department of Health • State of California Department of Health Services • State of Arizona Department of Health Services • US Army Corps of Engineers 	Section 11.1 Section 11.2.1	Sat			

COMPOSITE AUDIT CHECKLIST

QUALITY ASSURANCE

Organization:		Location:				Evaluation Date(s)
	Attributes	References Paragon LQAP	Sat	Un-sat	N/A	Comments
35.	Verify the following licenses, accreditation and certifications are held and maintained as applicable to Washington Group's subcontract: <ul style="list-style-type: none"> State of Colorado Department of Health State of Utah Department of Health State of California Department of Health Services State of Arizona Department of Health Services 	Section 11.3	Sat			
36.	Review and verify nonconformance reports. Are NCR's sequentially numbered and tracked on a tracking log?	Section 13.1		Un-Sat		Reference AFR No. 5
37.	Verify that NCR's are reviewed and approved by the analysis group supervision and Quality Assurance.	Section 13.1	Sat			
38.	Verify that out-of-control events are monitored against laboratory and project specific QA/QC requirements. Additionally when an event is determined to be out of control, verify that that laboratory initiates the appropriate level of corrective action to preclude future recurrence.	Section 13.2		Un-Sat		Reference AFR No. 7
39.	Are laboratory personnel trained commensurate with their duties, position, and responsibilities?		Sat			
40.	Review and verify that Paragon participates in inter-laboratory evaluation programs as sponsored by the following agencies: <ul style="list-style-type: none"> US EPA Water Pollution and Water Supply Study Audit Program State of California Department of Health Services Hazardous Waste PE Program Department of Energy (DOE), Office of Environmental Management (OEM) Quality Assessment Program EPA National Exposure Research Laboratory Characterization Research Division Environmental Resource Associates Proficiency Testing Program (quarterly) 	Section 11.2.1	Sat			



COMPOSITE AUDIT CHECKLIST

QUALITY ASSURANCE

Organization:		Location:			Evaluation Date(s)	
	Attributes	References Paragon LQAP	Sat	Un-sat	N/A	Comments
41.	<p>Review and verify that training records for all analytical staff members are being documented and maintained. Ensure that training records include the following as a minimum:</p> <ul style="list-style-type: none"> Records of academic training pertinent to the employees work assignment Summaries of training seminars attended while employed at Paragon Results of comprehensive testing or training Results of Health and Safety instruction received at Paragon Results of proficiency demonstrations as specified in Section 14.2.2 of the LQAP Current resume if available 	Section 14.3		Un-Sat		Reference AFR No. 2
42.	<p>Review and verify that the laboratory waste disposal program. Verify the classification of waste generated by Paragon Laboratory e.g., Small Quantity Waste Generator (SQWG) or large quantity waste generator.</p>	Section 16.1		Un-Sat		Reference AFR No. 1
43.	<p>Verify that Chain-of Custody/sample security requirements include:</p> <ul style="list-style-type: none"> Sample receipt requirements Sample verification Sample log-in requirements 	Section 5.2	Sat			
44.	<p>Visually inspect the waste storage area. Ensure the following:</p> <ul style="list-style-type: none"> Waste is labeled hazardous or non-hazardous Containers labeled type, start time, waste stream Satellite accumulation area is emptied frequently Containers have secondary containment 	Section 16.2	Sat			

**Washington****WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT****AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 01****Page 1 of 2**

ACTIVITY: Environmental Laboratory Audit

CLIENT: U.S EPA Response Action
Contract (RAC)

ORGANIZATION: Paragon Analytics Incorporated

REPLY DUE DATE: 7/8/01

STATEMENT OF REQUIREMENTS: Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, Section 1.5.1 states, "The LQAP is main guidance document for laboratory operations when there exists no other project or program-specific requirements to which the laboratory must conform. This document will be reviewed and updated at a minimum frequency of once every two years or more frequently if there are significant changes in procedures or capabilities in the laboratory."

FINDING: Contrary to the above requirements: See Attached Page 2Finding Classification: ☐ Major ☒ Minor PAAA Reportable Yes ☐ No ☒**RECOMMENDED CORRECTIVE ACTION:** See attached page 2.

You are requested to further investigate the finding(s) to identify the cause and effect of the condition(s) in order to determine the extent of corrective action required. The results of the investigation are to be considered in your reply.

AUDITOR:

Paul M. Ben

DATE:

*08/16/01***CORRECTIVE ACTION RESPONSE:**

(Attach additional sheets as necessary)

A. Action taken/proposed to correct findings:

B. Cause of Condition and Corrective Action to prevent recurrence:
Cause:

Corrective Action:

C. Completion Dates: (A: _____) (B: _____)

SIGNATURE _____ TITLE _____ DATE _____

EVALUATION OF RESPONSEAccept ☐
Reject ☐

SIGNATURE/TITLE _____

DATE _____

VERIFICATION OF IMPLEMENTATIONAccept ☐
Reject ☐
Not Required ☐

SIGNATURE/TITLE _____

DATE _____



FINDING: Contrary to the above requirements, it was determined that:

1. Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, has not been revised since February 1999. The Paragon Analytics, Laboratory Quality Assurance Plan has not been updated or revised since February 1999 which exceeds the minimum review and updated frequency as specified in the LQAP. During the course of the audit, Washington Group had noted many discrepancies between what was stated in the LQAP and what is currently being practiced in the laboratory.
2. The following discrepancies were noted:

Paragon Analytics LQAP Revision 4 Section 16.2 – Laboratory Waste Disposal

Waste Storage: "Paragon is classified as a small quantity generator, and generates between 100kg and 1000 kg of waste per month. Because of this rate of waste generation, waste materials created at the laboratory may accumulate on the site for a maximum of nine months, depending upon location of the Temporary Storage and Disposal Facility." Contrary to this requirement, Paragon's waste generator classification has changed from a small quantity generator to now a large quantity waste generator, which is not accurately reflected in Section 16.2 of the LQAP.

Paragon Analytics LQAP Revision 4 Section 15.1 – Laboratory Safety

Health and Safety Training – "The goal of Health and Safety (H&S) training is to ensure that the laboratory personnel have adequate knowledge to safely perform their assigned duties. This training is presented by laboratory's H&S Officer. Health and Safety training is provided to each employee as soon as possible after beginning work. The components of this course include, but are not limited to the following:

- An explanation of the Medical Surveillance Program, which includes annual physical for all employees engaged in laboratory activities."

Standard Operating Procedures LQAP Revision 4, Section 1.5.2

"Standard Operating Procedures (SOPs) are documents that describe in detail how laboratory procedures will be performed by the staff. SOPs will be reviewed and updated at a minimum frequency of once every two years or more frequently if there are significant changes (e.g., SW-846 update)."

Contrary to the above requirement, biannual updates or revisions to the following Standard Operating Procedures were not revised at the minimum biannual frequency as specified:

SOP 409, Revision 0, dated 02/15/1999– Analysis of Polychlorinated Biphenyls (PCBs) By Gas Chromatography – Method 8082

SOP 525, Revision 4, dated 02/12/1999 – Determination of Volatile Compounds By Gas Chromatography/Mass Spectrometry – Method 8260B and Method 624

RECOMMENDED CORRECTIVE ACTION:

Paragon Analytics Inc. should revise the LQAP to reflect the current manner in which business is being conducted in the laboratory. Standard Operating Procedures should also be revised in a timely manner. Since the LQAP is the basic document that represents an overview of laboratory functions, these procedural protocols should accurately reflect the methodologies used throughout the laboratory.

**Washington****WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT****AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 02****Page 1 of 2**

ACTIVITY: Environmental Laboratory Audit

CLIENT: U.S EPA Response Action
Contract (RAC)

ORGANIZATION: Paragon Analytics Incorporated

REPLY DUE DATE: 7/8/01

STATEMENT OF REQUIREMENTS: Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, Section 14.3 Training Records states, "Training records for all staff members will be maintained by the Paragon Quality Assurance Department. Training files may contain (but are not limited to) the following information:

1. Records of academic training pertinent to the employee's work assignment
2. Summaries of any training seminars attended while employed at Paragon
3. Any test results for examinations taken at Paragon
4. Records of Health & Safety instruction received while at Paragon
5. If available, a current resume of the employee"

FINDING: Contrary to the above requirements: See Attached Page 2Finding Classification: ☐ Major ☒ Minor PAAA Reportable Yes ☐ No ☒**RECOMMENDED CORRECTIVE ACTION:** See attached page 2.

You are requested to further investigate the finding(s) to identify the cause and effect of the condition(s) in order to determine the extent of corrective action required. The results of the investigation are to be considered in your reply.

AUDITOR:

Paul M. Ben

DATE:

*08/01/01***CORRECTIVE ACTION RESPONSE:**

(Attach additional sheets as necessary)

A. Action taken/proposed to correct findings:

B. Cause of Condition and Corrective Action to prevent recurrence:
Cause:

Corrective Action:

C. Completion Dates: (A: _____) (B: _____)

SIGNATURE _____ TITLE _____ DATE _____

EVALUATION OF RESPONSEAccept ☐
Reject ☐

SIGNATURE/TITLE _____

DATE _____

VERIFICATION OF IMPLEMENTATIONAccept ☐
Reject ☐
Not Required ☐

SIGNATURE/TITLE _____

DATE _____



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT**

**AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 02**

Page 2 of 2

FINDING: Contrary to the above requirements, it was determined that:

There is no objective evidence that Paragon Laboratory personnel have received laboratory department specific training or checklist thereof. Additionally, credentials attesting to the education, qualifications, and resumes of various staff personnel were either missing or incomplete. Further review of training records indicated that laboratory analysts/ technicians do not have documentation on file indicating that they have completed LQAP training, RCRA Waste training, etc. U.S Environmental Protection Agency Method SW-846 8000B mandates that the results of an analysts initial proficiency demonstration be posted to the individual training file or included in training records.

RECOMMENDED CORRECTIVE ACTION:

Washington Group International, Inc. Response Action Contract in support of the U.S. EPA mandates strict compliance to EPA Methods and laboratory protocols. Training records should be updated to document training proficiencies, and the results of training proficiencies included in each analyst file. In general, training records provide the necessary assurance that laboratory personnel are trained, qualified and that they are proficient at their assigned task. Paragon Laboratory QA Manager should assess all training records and update all personnel training files as specified in LQAP Section 14.2.2.2 and SW-846 8000B.



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT**

**AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 03**

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ACTIVITY: Environmental Laboratory Audit

CLIENT: U.S EPA Response Action
Contract (RAC)

ORGANIZATION: Paragon Analytics Incorporated

REPLY DUE DATE: 7/8/01

STATEMENT OF REQUIREMENTS: Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, Section 9.0 Quality Control Procedures states, "A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out of control events, and requiring corrective action measures to prevent or minimize the recurrence of these events." EPA Method 8000B Determinative Chromatographic Separations paragraph 8.7.5 states, "Once established, control limits and warning limits for spike compounds should be reviewed after every 10 – 20 matrix spike samples of the same matrix, and updated at least semi-annually. Control limits and warning limits for surrogates should be reviewed after every 20 – 30 field samples of the same matrix, and should be updated at least semi annually. The laboratory should track trends in both performance and in the control limits themselves. The control and warning limits used to evaluate the sample results should be those in place at the time the sample was analyzed. Once limits are updated, those limits should apply to all subsequent analyses of new samples."

FINDING: Contrary to the above requirements: See Attached Page 2

Finding Classification: ☐ Major ☒ Minor PAAA Reportable Yes ☐ No ☒

RECOMMENDED CORRECTIVE ACTION: See attached page 2.

You are requested to further investigate the finding(s) to identify the cause and effect of the condition(s) in order to determine the extent of corrective action required. The results of the investigation are to be considered in your reply.

AUDITOR:

Paul M. Ben

DATE:

08/04/01

CORRECTIVE ACTION RESPONSE:

(Attach additional sheets as necessary)

A. Action taken/proposed to correct findings:

B. Cause of Condition and Corrective Action to prevent recurrence:
Cause:

Corrective Action:

C. Completion Dates: (A: _____) (B: _____)

SIGNATURE _____ TITLE _____ DATE _____

EVALUATION OF RESPONSE

Accept ☐
Reject ☐

SIGNATURE/TITLE

DATE

VERIFICATION OF IMPLEMENTATION

Accept ☐
Reject ☐
Not Required ☐

SIGNATURE/TITLE

DATE



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT**

**AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 03**

Page 2 of 2

FINDING: Contrary to the above requirements, it was determined that:

Internal Paragon Laboratory Quality Control Procedures were reviewed to determine the in-house systematic process controls implemented to measure and detect errors or out-of-control events. In-house quality controls are defined and implemented through various procedures. The criterion that is used to measure and analyze environmental data includes measurements of accuracy and precision. However, control limit measurements that are required to reflect the degree to which the measured value approximates the actual or true value for a given parameter. The control limits, which influence bias in measurements, are not being updated semi-annually or annually for some methods as required by EPA Method Protocols.

RECOMMENDED CORRECTIVE ACTION:

Washington Group International, Inc. Response Action Contract in support of the U.S. EPA mandates strict compliance to EPA Methods and laboratory protocols. The control limits, which influence bias in measurements, should be updated semi-annually or annually as required by EPA Method Protocols. In general, process controls provide the necessary assurance that laboratory processes can measure and detect out-of control events. Paragon Laboratory QA Manager should update all applicable control limit measurements as specified in LQAP and SW-846 8000B.



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT**

**AUDIT NO.: RAC-V-01-01,
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ACTIVITY: Environmental Laboratory Audit

CLIENT: U.S EPA Response Action
Contract (RAC)

ORGANIZATION: Paragon Analytics Incorporated

REPLY DUE DATE: 7/8/01

STATEMENT OF REQUIREMENTS: Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, Section 8.0, Preventative Maintenance states, "The objective of Paragon's preventative maintenance program is to establish a system of instrument care that prevents the loss of analytical quality control and results in the minimum of lost productivity due to instrument failure."

FINDING: Contrary to the above requirements: See Attached Page 2

Finding Classification: ☐ Major ☒ Minor PAAA Reportable Yes ☐ No ☒

RECOMMENDED CORRECTIVE ACTION: See attached page 2.

You are requested to further investigate the finding(s) to identify the cause and effect of the condition(s) in order to determine the extent of corrective action required. The results of the investigation are to be considered in your reply.

AUDITOR:

Paul M. Bon

DATE:

08/16/01

CORRECTIVE ACTION RESPONSE:

(Attach additional sheets as necessary)

A. Action taken/proposed to correct findings:

B. Cause of Condition and Corrective Action to prevent recurrence:
Cause:

Corrective Action:

C. Completion Dates: (A: _____) (B: _____)

SIGNATURE _____ TITLE _____ DATE _____

EVALUATION OF RESPONSE

Accept ☐
Reject ☐

SIGNATURE/TITLE _____

DATE _____

VERIFICATION OF IMPLEMENTATION

Accept ☐
Reject ☐
Not Required ☐

SIGNATURE/TITLE _____

DATE _____



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT**

**AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 04**

Page 2 of 2

FINDING: Contrary to the above requirements, it was determined that:

During the course of the audit, a GC/MS pump and GC OI Purge and Trap located in a laboratory were observed as being set off to the side. Careful examination of the instrumentation indicated that it was not in use and/or it was broken. Further investigation revealed that the item was not properly tagged indicating it's operating status as required by Paragon SOP 319.

RECOMMENDED CORRECTIVE ACTION:

The Washington Group International, Inc. audit team recommends that the appropriate tags be place on instrumentation or equipment that is placed out of service, broken or malfunctioning. Additionally, instrumentation should be placed in a designated area that is segregated from all other instrumentation to prevent inadvertent placement of the instrumentation into service or inadvertent use.



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
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**AUDIT NO.: RAC-V-01-01,
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ACTIVITY: Environmental Laboratory Audit

CLIENT: U.S EPA Response Action
Contract (RAC)

ORGANIZATION: Paragon Analytics Incorporated

REPLY DUE DATE: 7/8/01

STATEMENT OF REQUIREMENTS: Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, Section 12.0, Quality Assurance Reports to Management states, "For day-to-day reporting, A Nonconformance Report (NCR) is initiated for laboratory QA situations that require immediate attention. The employee that discovers the nonconformance is responsible for initiating the NCR. The Project Manager and QA Manager must approve the corrective action proposed." Section 13.1 Nonconformance Report further states, "Nonconformance Reports (NCRs) are controlled documents that are administered by Paragon's Quality Assurance Group. The staff member will then complete the form by entering all pertinent information and the final disposition required to adequately address the Non-Conformance".

FINDING: Contrary to the above requirements: See Attached Page 2

Finding Classification: ☐ Major ☒ Minor PAAA Reportable Yes ☐ No ☒

RECOMMENDED CORRECTIVE ACTION: See attached page 2.

You are requested to further investigate the finding(s) to identify the cause and effect of the condition(s) in order to determine the extent of corrective action required. The results of the investigation are to be considered in your reply.

AUDITOR:

Paul M. Burr

DATE:

08/16/01

CORRECTIVE ACTION RESPONSE:

(Attach additional sheets as necessary)

A. Action taken/proposed to correct findings:

B. Cause of Condition and Corrective Action to prevent recurrence:
Cause:

Corrective Action:

C. Completion Dates: (A: _____) (B: _____)

SIGNATURE _____ TITLE _____ DATE _____

EVALUATION OF RESPONSE

Accept ☐
Reject ☐

SIGNATURE/TITLE _____

DATE _____

VERIFICATION OF IMPLEMENTATION

Accept ☐
Reject ☐
Not Required ☐

SIGNATURE/TITLE _____

DATE _____



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT**

**AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 05**

Page 2 of 2

FINDING: Contrary to the above requirements, it was determined that:

During the course of this audit, reports to management were verified by review of nonconformance reports. The audit team reviewed various nonconformance reports and corresponding dispositions. Routine NCR dispositions, such as use as is, reject and/or repair, are not marked on the NCR form. Objective evidence of the disposition process indicated that in many cases the disposition was recorded as "Document in a Narrative". Further investigation indicated that in most cases, the narrative is undefined and is not attached or part of the disposition and closure of the NCR.

The NCR system does not provide adequate confidence that the nonconformance reporting and subsequent corrective actions are being disposition to preclude recurrence and are being tracked from initiation through closure.

RECOMMENDED CORRECTIVE ACTION:

The Washington Group International, Inc. audit team recommends that Nonconformance reports include those documents e.g., Documented Narratives to be included in the final resolution/disposition and corrective action verification of nonconformance reports.

**Washington****WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT****AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 06****Page 1 of 2**

ACTIVITY: Environmental Laboratory Audit

CLIENT: U.S EPA Response Action
Contract (RAC)

ORGANIZATION: Paragon Analytics Incorporated

REPLY DUE DATE: 7/8/01

STATEMENT OF REQUIREMENTS: Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, Section 11.0, Performance and System Audits states, "Two types of internal audit procedures will be used to assess and document performance of laboratory staff: systems audits and performance evaluation audits." Section 11.1.1 Internal Systems Audits states, "This audit is general in nature, and provides an overview of laboratory operations. This type of audit must be performed at least once a month unless an external audit is performed during the same calendar month. The laboratory QA Manager will perform the laboratory system audit in accordance with checklists designed to aid the auditor in ensuring that all areas of laboratory operations are reviewed." Section 11.1.1 further states... "Audit results are reported in writing to responsible management for review and corrective action if necessary. A maximum of two weeks is given to respond to the original report."

FINDING: Contrary to the above requirements: See Attached Page 2Finding Classification: ☐ Major ☒ Minor PAAA Reportable Yes ☐ No ☒**RECOMMENDED CORRECTIVE ACTION:** See attached page 2.

You are requested to further investigate the finding(s) to identify the cause and effect of the condition(s) in order to determine the extent of corrective action required. The results of the investigation are to be considered in your reply.

AUDITOR: *Pat M. Ben*DATE: *08/14/01***CORRECTIVE ACTION RESPONSE:**

(Attach additional sheets as necessary)

A. Action taken/proposed to correct findings:

B. Cause of Condition and Corrective Action to prevent recurrence:
Cause:

Corrective Action:

C. Completion Dates: (A: _____) (B: _____)

SIGNATURE _____ TITLE _____ DATE _____

EVALUATION OF RESPONSEAccept ☐
Reject ☐

SIGNATURE/TITLE _____

DATE _____

VERIFICATION OF IMPLEMENTATIONAccept ☐
Reject ☐
Not Required ☐

SIGNATURE/TITLE _____

DATE _____



FINDING: Contrary to the above requirements, it was determined that:

During the course of this audit, a schedule of audits and corresponding audit reports were reviewed. However, the audit schedule indicated that scheduled examinations of the operations of specific analytical departments were logged as being initiated, but were never formally closed or subsequent corrective actions taken or noted. Additionally, the specified performance frequency (e.g., once per month) in many cases is being exceeded by two or three month intervals. Careful examinations of the audits that have been initiated to date clearly indicate that the evaluation and implementation of specific quality related systems should be improved. The following internal audits were log as being initiated or performed, however the audit report and subsequent corrective actions were not available for review:

Audit No.	Department	Date
IA12199	GC Fuels	01/31/00
IA032000	Metals Rad	04/17/00
	GC SVOC M8081A	06/12/00
	GC SVOC M8082	06/17/00
SR07100	Internal C of C	07/31/00
Unknown	GC/MS/VOC	08/16/00
Unknown	GC Fuels Instrument PC & Backup	09/28/00
Unknown	Organic Extractions Prep & Analysis	10/16/00

In addition, SOP-937 Revision 2, paragraph 2.2, Internal Laboratory Audits specifies that audits will be performed by designated staff, which may or may not use an auditing aid such as checklists. The laboratory audits that were reviewed did not include checklists.

RECOMMENDED CORRECTIVE ACTION:

The Washington Group International, Inc. audit team recommends that Performance Audits be conducted at the intervals specified in Section 11.1.1 of Paragon's LQAP. If internal laboratory audits can not be performed or scheduled as specified in the LQAP, then the LQAP should be revised to accommodate a more flexible schedule. Additionally, the requirement specified in LQAP section 11.1.1 and SOP 937 contradict. The audit team recommends to use checklists as specified or revise the LQAP to be more compatible with the requirements specified in SOP 937. Please provide in your response corrective actions taken to preclude recurrence.



Washington

**WASHINGTON GROUP QUALITY
ASSURANCE
AUDIT FINDING REPORT**

**AUDIT NO.: RAC-V-01-01,
Rev. 1
AFR No.: 07**

Page 1 of 2

ACTIVITY: Environmental Laboratory Audit

CLIENT: U.S EPA Response Action
Contract (RAC)

ORGANIZATION: Paragon Analytics Incorporated

REPLY DUE DATE: 7/8/01

STATEMENT OF REQUIREMENTS: Paragon Analytics, Laboratory Quality Assurance Plan Revision 4, dated 02/99, Section 13, Corrective Actions states, "Corrective action is necessary when any measurement system fails to follow this LAQP... In general, items needing corrective action fall into two "correction categories" short term and long term. Long Term Corrective Actions The actions consist of minor and major problems which require a series of actions to resolve the problem. The actions to be taken are coordinated by the Section Manager or QA Manager, and a Non Conformance Report (Appendix D) is used to document the action. The report will describe the analysis involved, the date, analyst, the identification of all affected or suspect samples, probable cause, the corrective action measure(s) taken, and the final disposition/resolution of the problem."

FINDING: Contrary to the above requirements: See Attached Page 2

Finding Classification: ☐ Major ☒ Minor PAAA Reportable Yes ☐ No ☒

RECOMMENDED CORRECTIVE ACTION: See attached page 2.

You are requested to further investigate the finding(s) to identify the cause and effect of the condition(s) in order to determine the extent of corrective action required. The results of the investigation are to be considered in your reply.

AUDITOR:

P. M. B.

DATE:

08/16/01

CORRECTIVE ACTION RESPONSE:

(Attach additional sheets as necessary)

A. Action taken/proposed to correct findings:

B. Cause of Condition and Corrective Action to prevent recurrence:
Cause:

Corrective Action:

C. Completion Dates: (A: _____) (B: _____)

SIGNATURE _____ TITLE _____ DATE _____

EVALUATION OF RESPONSE

Accept ☐
Reject ☐

SIGNATURE/TITLE _____

DATE _____

VERIFICATION OF IMPLEMENTATION

Accept ☐
Reject ☐
Not Required ☐

SIGNATURE/TITLE _____

DATE _____



FINDING: Contrary to the above requirements, it was determined that:

During the course of this audit, a schedule of audits and corresponding audit reports were reviewed. However, the audit schedule indicated that scheduled examinations of the operations of specific analytical departments were logged as being initiated, but were never formally closed or subsequent corrective actions taken or noted. Additionally, the specified performance frequency (e.g., once per month) in many cases is being exceeded by two or three month intervals. Careful examinations of the audits that have been initiated to date clearly indicate that the evaluation and implementation of specific quality related systems should be improved. The following internal audits were logged as being initiated or performed, however the audit report and subsequent corrective actions were not available for review:

Audit No.	Department	Date
IA12199	GC Fuels	01/31/00
IA032000	Metals Rad	04/17/00
	GC SVOC M8081A	06/12/00
	GC SVOC M8082	06/17/00
SR07100	Internal C of C	07/31/00
Unknown	GC/MS/VOC	08/16/00
Unknown	GC Fuels Instrument PC & Backup	09/28/00
Unknown	Organic Extractions Prep & Analysis	10/16/00

In addition, SOP 937 Revision 2, paragraph 2.2, Internal Laboratory Audits specifies that audits will be performed by designated staff, which may or may not use an auditing aid such as checklists. The laboratory audits that were reviewed did not include checklists.

RECOMMENDED CORRECTIVE ACTION:

The Washington Group International, Inc. audit team recommends that corrective actions of audit deficiencies be formulated for and closed for the items noted above. If internal laboratory audits are scheduled but can not be performed as scheduled then the audit log should annotate that the audit could not be performed. Additionally, corrective actions to audit deficiencies are to be reported to management for review, the above noted audits were logged as being completed. However, records could not substantiate if the appropriate corrective actions were reviewed verified and effectively implemented. Additionally, the requirement specified in LQAP Section 11.1.1 and SOP 937 contradict. The audit team recommends the use of checklists, as specified, or revise the LQAP to be more compatible with the requirements specified in SOP 937. Please provide in your response corrective actions taken to preclude recurrence.



AUDIT OBSERVATION REPORT

AOR No.:1
AUDIT No.: RAC-V-01-01, Rev.1

ACTIVITY: Analytical Laboratory Audit CLIENT: U.S. Environmental Protection Agency
ORGANIZATION: Paragon Analytics Inc.

STATEMENT OF REQUIREMENTS:

Paragon Analytics Laboratory Quality Assurance Program Revision 4, Section 17.1 Receipt Verification of Standards states, "All primary reference standard and standard solutions are purchased from reliable commercial sources. Standards traceable to NIST are preferred; however, ASTM or equivalent specifications are acceptable. Certification records of all standards received are retained."

Section 17.2 Receipt Verification of Solvents and Acids states "The verification procedure for organic solvents involves taking an initial volume of solvent and concentrating it to a reduced final volume. The initial volume used for this procedure and its final volume vary depending upon solvent..."

OBSERVATION

A review of various Purchase Orders indicated that quality related or quality affecting items do not receive quality assurance review. Purchase Order Number 001869 and P.O. 23867 was reviewed. During review it was noted that the items being purchased were not reviewed or approved.

Contrary to the above requirement the audit team could not verify that a solvent testing program is currently in place as specified in section 17.2 of the LQAP.

Classification: Major ☒ Minor ☐ Response Due Date: 07/08/01

AUDITOR

Paul M. Burr

DATE

08/16/01

OBSERVATION RESPONSE Major Observations only

SIGNATURE

TITLE Lead Auditor

DATE :



AUDIT OBSERVATION REPORT

AOR No.:2

AUDIT No.: RAC-V-01-01, REV.1

ACTIVITY: Analytical Laboratory Audit CLIENT: U.S. Environmental Protection Agency

ORGANIZATION: Paragon Analytics Inc.

STATEMENT OF REQUIREMENTS:

The following observations were made of laboratory practices that of noteworthy. No response is required.

OBSERVATION

Monthly supervisory reviews of laboratory logbooks are not being performed on a consistent basis

The small hood in the GC laboratory is being used for standard preparation when it is only designed for nuisance odor use.

Classification: Major ☐ Minor ☒ Response Due Date: N/A No Response Required

AUDITOR

Palm. Ben

DATE

08/16/01

OBSERVATION RESPONSE Major Observations only

N/A No response Required

SIGNATURE

TITLE Lead Auditor

DATE :



AUDIT OBSERVATION REPORT

AOR No.:3

AUDIT No.: RAC-V-01-01, REV.1

ACTIVITY: Analytical Laboratory Audit CLIENT: U.S. Environmental Protection AgencyORGANIZATION: Paragon Analytics Inc.**STATEMENT OF REQUIREMENTS:**

Paragon Analytics Laboratory Quality Assurance Program Revision 4, Section 10 Data Reduction, Validation and Reporting states "During the course of processing and reviewing sample analysis results, it may be necessary to correct documentation errors discovered during this process. To maintain the integrity of the documentation generated by the laboratory in order to meet potential litigation requirements, changes to documents must be made in the following manner:

1. A single line will be struck through the entry to be changed
2. A new entry with the correct information will be made;
3. The date the change was made will be recorded; and;
4. The initials of the person making the change will be entered."

Section 10.4 Data Validation states, "All analytical data generated by Paragon Analytics, Inc. are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review."

OBSERVATION

Work Order No. 0103075 was reviewed. During the audit team's review the following observations were noted:

- Many organic laboratories were missing the annotation of the amounts of various standards added to samples during prep or analysis on the run log books
- Corrections to sample extraction and preparation laboratory worksheets for ignitability or GC pesticide data were not corrected with a single line through and initial and date.
- The analysts improperly documented manual integration. A review of GC/MS SVOA and Pesticide data indicated that the "before and after" reason for integration and subsequent initial and date are missing.
- Case narratives are incomplete. A review of GC/MS SVOA narrative revealed that dilutions were initiated for WGI samples. However, the case narrative did not provide an explanation or reason as to why the dilutions were necessary, and an explanation ascertaining why undiluted samples did not have target compounds over the linear range.

Contrary to the above requirements, the audit teams observations were noteworthy in the identification of generic areas that are in need of improvement. No response required.

Classification: Major ☐ Minor ☒ Response Due Date: N/A

AUDITOR

DATE

08/16/01

OBSERVATION RESPONSE Major Observations only

N/A – No Response Required

SIGNATURE

TITLE Lead Auditor

DATE :

ENCLOSURE 2:

External Audit Reports / Paragon Responses

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Los Alamos National Laboratory

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ESH-17, Air Quality Group
P.O. Box 1663, Mail Stop J978
Los Alamos, New Mexico 87545
(505) 665-8855 / FAX: (505) 665-8858

Date: June 27, 2001
Refer to: ESH-17:01-314

Ms. Debra Henderer
Quality Assurance Officer
Paragon Analytics, Inc.
225 Commerce Drive
Fort Collins, CO 80524

Dear Debra:

Enclosed is the final version of our CY 2000 supplier assessment report resulting from my on-site visit last October. Thank you and all of the fine staff at Paragon for their cooperation with this portion of our Quality Management Program and their continuing high quality analytical work on our behalf.

Sincerely,



Ernest S. Gladney, Ph.D.
Air Quality Group

ESG:db

Enc: a/s

Cy: (w/enclosure)
Terry Morgan, ESH-17, J978
Jean Dewart, ESH-17, J978
Scott Miller, ESH-17, J978
Craig Eberhart, ESH-17, J978
Dave Fuehne, ESH-17, J978
ESH-17 File

LANL ESH-17 Assessment of the Radiochemistry Facilities
at
Paragon Analytics, Inc. (PAI)
16-17 October 2000

Executive summary

One finding and no new observations resulted from this assessment and are described in this report. Significant progress on procedure (SOP) review and update has been made since the previous assessment. Additionally, significant progress has been made in the implementation and maintenance of the full quality program in keeping with the excellent technical performance of the organization. The quality management program is now fully operational.

Scope of the assessment

Paragon Analytics, Inc. (PAI), currently provides beryllium determination on stack filters and liquid scintillation counting of distillate from silica-gel-cartridge tritium-in-air samples collected at LANL ESH-17 AIRNET ambient air sampling stations. Recently they have begun to conduct the distillation of ambient atmospheric moisture directly from silica gel samples submitted to them. They also serve as the ESH-17 referee laboratory whenever high sample results from another vendor necessitate chemical analysis of the remaining filter fraction. Additionally, there is the potential that PAI could provide additional radioanalytical services of air-sample media in the future. This assessment concentrated on the technical capabilities and operation of the radiochemistry and inorganic laboratories, but included a follow-up review of the Paragon Analytics quality program, last assessed by ESH-17 in Aug. 1999.

Assessors

Dr. Ernest S. Gladney, the ESH-17 lead assessor and analytical chemistry coordinator, conducted the entire assessment and this report, including the actual site visit.

Assessment schedule

The assessment was conducted on Monday and Tuesday, 16-17 October 2000. The assessment began with an in-brief meeting attended by:

Don Gipple, President/Laboratory Director
Lori Pacheco, Operations Manager
Deb Henderer, Quality Assurance Manager
Steve Workman, Inorganics Technical Manager
Dave Burns, Radiochemistry Operations Manager
Darryl Patrick, Inorganics Supervisor

The results of the assessment were discussed at a close-out meeting attended by:

Don Gipple Lori Pacheco
Deb Henderer Steve Workman
Darryl Patrick
Steve Fry, Vice President
Anthony Vargrees, Rad Chem Technical Manager
Scott Hafeman, Radiochemist

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Conduct of the assessment

A general checklist, based on Clean Air Act requirements and industry-standard radiochemistry practices, was used as a guide for this assessment. However, the inquiry was not limited to the checklist and relied heavily on the professional experience of the assessor. The assessment methods included document and record reviews, personnel interviews, and laboratory facility inspections.

Definitions

Finding – An area of operation or function described in Paragon or ESH-17 documents that was found not implemented or an area or technical function directly impacting either a Statement of Work issue or the credibility of Paragon analysis data used by ESH-17 for compliance reporting. ESH-17 requests documented proposed and final corrective actions for findings.

Observation – An area of operation or function described in Paragon or ESH-17 documents or a recommended practice that was found to be less than fully implemented or an area or function that *could* directly impact the credibility of analysis data used by ESH-17 for compliance reporting. . Suggestions for improved customer-supplier communications also fall within this area. Formal documented corrective actions are not required or requested

Noteworthy practices

In the previous ESH-17 assessment in 1999, two noteworthy practices were identified. The following noteworthy practices were identified during the present assessment:

1. Disposal of Remnants of Environmental Samples

A new dedicated facility has been constructed within the Paragon laboratory building, dedicated to the proper classification and disposal of the unconsumed portions of environmental samples submitted from the company's various customers. An excellent and responsible system is being put in place for sorting these remnants into appropriate waste streams, and a knowledgeable technician is running this process.

2. Continuing Excellent Performance in External Blind Proficiency Testing

Although hard to construe as a noteworthy practice, *per se*, Paragon's exceptional performance in external blind proficiency testing over the past several years is, nevertheless, a noteworthy fact, which indicates excellent measurement capability. This high performance level during the past year occurs in both radiological and nonradiological performance measurements.

Status of Prior ESH-17 Findings

One finding from the Nov. 1998 assessment had been carried over as incompletely addressed: The status of that finding was evaluated during the present assessment.

1. No analytical procedure training records for Paragon employee performing analyses on ESH-17 samples.

Status—Ample evidence was presented regarding resolution of this very specific finding. Therefore, this unresolved prior finding is now **RESOLVED**.

During the Aug. 1999 assessment, one new finding was identified. The status of that finding was evaluated during the present assessment.

1. Most radiochemistry procedures are overdue for review

Status – Most Paragon analytical procedures have undergone review and updating during the past year. Only five radiochemistry ones remain to be completed, none of which apply to work being done for ESH-17. This prior finding is now **RESOLVED**.

New Finding

1. Beryllium determinations on stack filters submitted by ESH-17 are not reported with any indication of the uncertainty in the measurement

Requirement: The ESH-17 Statement of Work “General Requirements for all Statements of Work For Analytical Chemistry Support for ESH-17 (LANL/ESH-17/GEN, 01/01/2000 version)” states:

“ ... **ELECTRONIC DATA DELIVERABLE (EDD) ...**”

3. Summaries of sample results shall include: customer id, sample delivery group or request number, lab id, specific analysis by radionuclide or element as applicable, analyte concentration, analyte uncertainty and MDA in the same appropriate units, tracer recoveries (where appropriate in fractional percent), and dates of analysis.

4. Summaries of QA/QC results shall include the same parameters as sample results. ...

DATA PACKAGE DELIVERABLE ...

All hardcopy data packages shall include the following, at a minimum: ...

5. Summaries of sample results shall include: customer id, sample delivery group or request number, lab id, isotope/analysis, analyte concentration, analyte uncertainty and MDA in the same appropriate units, tracer recoveries (where appropriate in fractional percent), and dates of analysis.

6. Summaries of QA/QC results shall include the same parameters as sample results. ...”

Discussion: Agreement has been reached between ESH-17 and Paragon regarding how these uncertainties are to be estimated and reported. Spiked filter studies are in progress and based upon those results an estimated uncertainty calculation procedure will be implemented at ESH-17.

Unresolved Prior Observations from 1998

During the November 13, 1998 assessment, eight observations were identified. The status of those observations was evaluated during the present assessment.

1. Procedure 704 has not been reviewed or updated since 1994

Procedure has been recently revised. **CLOSED**

3. Not all maintenance and repair work performed on the Beckman liquid scintillation counting instruments has been recorded in instrument logbooks

Maintenance logbook now contains complete records of repair work. **CLOSED**.

4. Records for distribution of controlled copies of the LQAP are incomplete

Distribution process clarified. Controlled copies are sent to analytical groups but not to individuals. **CLOSED**.

5. Wet chemistry sample storage room was overcrowded and secondary containment of samples was not used for many liquid samples

Conditions appeared to be good on the day observed. However, some samples are still not stored in secondary containment. **REMAINS only PARTIALLY RESOLVED**.

6. QA department is not maintaining employee training files per the LQAP

This process has been clarified and implemented. **CLOSED**

Prior Observations from 1999

1. Retired procedures were still in both the master copy and the controlled copies of procedures.

Retired procedures have been removed. **CLOSED**

2. Housekeeping in halls and sample receipt area not well maintained.

This now appears to be addressed continuously. **CLOSED**

3. Section 1 of the LQAP and the detailed Table of Contents does not correspond.

Apparent conflict corrected. **CLOSED**

4. SOW LANL/ESH-17/GEN not being fully met in all cases.

A revised agreement was made that ESH-17 would obtain these data during each assessment. **CLOSED**

New Observations

None

Analytical Quality Control Performance**1. Quality control summary**

The quality control (QC) results discussed in this section apply to all of CY1999. Paragon has provided Laboratory Control Standards (LCS) and process blanks (PB), while ESH-17 has regularly submitted trip blanks (TB), matrix blanks (MB), and blind matrix spikes (MS). Minimum Detectable Activity (MDA) is determined on each sample. The following table summarizes results for CY 1999:

QC Type	Units	Mean	SD	# samples
LCS	% Recovery	99.0	3.3	120
MS	% Recovery	95.6	4.7	57
PB	pCi/mL	0.0	0.1	175
TB	pCi/mL	0.10	0.21	52
MB	pCi/mL	-0.04	0.16	78
MDA (samples)	pCi/mL	0.54	0.16	1339

Paragon has demonstrated an excellent record for LCS recovery and contamination control on all the various blanks. Overall MDA meets the statement of work (SOW) requirement of 2 pCi or 0.5 pCi/mL on 5 mL samples.

2. National analytical laboratory performance evaluation studies

Participation in both EPA, ERA (successor to EPA), DOE/EML and DOE/MAP national performance evaluation programs is required by the LANL ESH-17 SOW. Paragon participated in every available round. Their results reported in these programs during CY 1999 were judged "acceptable" in all cases. In general, "acceptable" performance represents achievement of analytical results that are within two standard deviations (SD) of the agency certified value (CV), "warning" represents analytical results that are between two and three SD from the CV, and "not acceptable" analytical results are outside three SD from the CV. Paragon participated successfully in all required national analytical laboratory performance evaluation programs for which we have currently received information, for the nuclides and inorganics of interest to ESH-17 during CY 1999.

Assessor conclusions regarding the Paragon Analytics radiochemistry program

1. Quality at Paragon

The technical quality of the analysis work performed for ESH-17 continues to be very good. Data packages continue to have very few discrepancies. As concluded in the previous assessment and as also apparent during this assessment, Paragon employees are knowledgeable, well trained, and enthusiastic about their work.

In my professional opinion, Paragon continues to be fully qualified to perform H-3 and Be determinations on air filter media for ESH-17.


Ernest S. Gladney, Ph.D.
Certified Quality Systems Lead Assessor

Date



PARAGON ANALYTICS, INC.

225 Commerce Drive ♦ Fort Collins, CO 80524 ♦ (800) 443-1511 ♦ (970) 490-1511 ♦ FAX (970) 490-1522

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Via Federal Express

September 15, 2000

Mr. David Carden
Environmental Management National Analytical Management Program
U.S. Department of Energy
Oak Ridge Operations
P.O. Box 2001
Oak Ridge, TN 37830

**Re: Department of Energy – Office of Environmental Management,
National Analytical Management Program
Environmental Management Consolidated Audit Program
Laboratory Qualification Audit of Paragon Analytics, Inc.
March 30-31, 2000
Paragon's Corrective Action Report**

Dear Mr. Carden:

I am writing to respond to your report regarding the March 30-31, 2000 on-site audit of Paragon Analytics, Inc., which I received on July 26, 2000. Paragon sincerely appreciates the on-site audit of our systems and processes and the time spent with our employees. We are pleased to respond to the 22 findings and 17 observations from the audit. Paragon's responses and corrective actions follow for your review.

Findings

Quality Assurance Management Systems

Item M1-000331-A: *The effectiveness of the Corrective Action implementation is not reviewed. (Priority II) (Integrated Contractor Procurement Team Basic Ordering Agreement (ICPT BOA), ICPT BOA, Attachments B and C, Criterion 3).*

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Response M1-000331-A:

In response to this finding, Paragon has reviewed the summary reports and close-out reports from the 14 internal audits performed since May 1999. We have determined that all prescribed corrective actions have been completed.

In response to the auditor's comments regarding Internal Audit IA51299, Paragon notes that the auditor was reading Revision 2 of SOP 202 (01/06/99), which had not been updated per IA51299. Revision 4 of SOP 202 (01/17/00) had been updated but had not yet been filed in the SOP binder. Copies of SOP 202, Revisions 2 and 4, are enclosed for your review (*Attachment M1-000331-A*).

Corrective Action	Date of Completion	Responsible Parties
Review summary reports and close-out reports from 14 IAs	08/29/00	QA Department

Finding M1-000331-B:

PAI is not opening coolers in or in the vicinity of an operable fume hood. (Priority II) (ICPT BOA, Attachment D)

Response M1-000331-B:

Paragon acknowledges that incoming packages were not opened in a fume hood, but in an open area of the laboratory, at the time of the audit. We attribute the root cause of this finding to the design of the Sample Control Laboratory, which precludes technicians from opening coolers in a fume hood.

In order to correct this deficiency, Paragon has developed short-term and long-term corrective actions. The short-term corrective action consists of purchasing a portable canopy hood that will be installed in the Sample Control Laboratory. This portable canopy hood will enable technicians to perform the initial inspection of coolers within a vented enclosure and will not require lifting of relatively heavy coolers. Following initial inspection, coolers that contain only intact samples will be removed and processing will continue outside the vented canopy hood. Any cooler that contains damaged samples will be lifted to the fixed fume hoods for further processing. Paragon has retained Installations Unlimited of Loveland, Colorado to design, build, and install the portable canopy hood. We anticipate that installation of this unit will be completed within 90-120 days.

The long-term corrective action involves structural additions to the building. In preparation, Paragon has purchased two (2) six-foot walk-in hoods that will be installed in the 35 ft x 70 ft addition to the Sample Control Laboratory (please see *Attachment M1-000331-B* for documentation of check #27307 to D. L. Chaney Scientific for the two hoods). These fixed fume hoods will enable technicians to open all coolers within a vented enclosure at floor level. Initial estimates suggest a total cost of approximately \$100k; therefore, dates of completion of the building addition and hood installation will be determined by financial considerations.

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Corrective Action	Date of Completion	Responsible Parties
Design, build, install portable canopy hood	12/31/00	Ed Wallace, Designs Unlimited of Loveland
Purchase two 6-foot walk-in hoods for structural addition	06/22/00	Ed Wallace
Complete structural addition; install hoods	TBD	Don Gipple

Item M1-000331-C: *PAI has not completed required reviews and updates of their SOPs. (Priority II) (ICPT BOA, Attachments B and C, Criterion 4; PAI QAP).*

Response M1-000331-C: Paragon acknowledges that we have not completed the review and revision of all SOPs within the past two years, per our QAP requirement. As of this writing, 195 SOPs (total) have been reviewed and revised since December 1998. Paragon has reviewed and revised 61 SOPs since the DOE ORO audit of December 6-7, 1999. Approximately 25 SOPs have not yet been reviewed and revised in the past two years. Paragon continues to work toward the goal of reviewing and revising every SOP within a two (2) year period. Paragon anticipates that these 25 SOPs will be reviewed and revised or retired by December 31, 2000. Attached for your review please find: (1) a table of contents for SOPs that demonstrates the latest date of review and revision and (2) the QA Department's documentation of distribution of controlled SOPs since December 1999 (*Attachment M1-000331-C*).

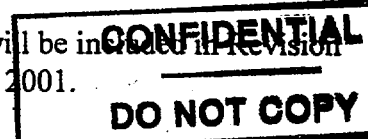
Corrective Action	Date of Completion	Responsible Parties
195 SOP updates completed (total)	08/31/00	QA Department
61 SOPs updates completed since DOE ORO audit 12/99	08/31/00	QA Department
25 SOP updates to be completed	12/31/00 (target)	QA Department

Item M1-000331-D: *PAI definition of training requirements and record maintenance have not been fully implemented (Priority II) (PAI QAP).*

Response M1-000331-D: Paragon acknowledges that the internal training program and documentation of training is incomplete. Paragon has made significant progress in defining training requirements and documenting training in the past 6 months and we continue to work toward full implementation. Following is a discussion of items that have been addressed by the QA Department.

General requirements for training are presented in the Laboratory Quality Assurance Plan (LQAP), Section 14. Paragon has revised these requirements as demonstrated in

Attachment M1-000331-D. Revisions shown in these pages will be included in Revision 5 of the LQAP, which is scheduled for publication in February 2001.



Paragon has updated, renamed, and released SOP 143, which is referred to in Section 14 of the LQAP and noted as non-existent by the auditors. This SOP formalizes and describes the QA orientation and training overview for new employees. A copy of Revision 1 is included with **Attachment M1-000331-D** for your review.

Paragon also includes sample pages from the QA Department's "Training Records Tracker" spreadsheet that demonstrates how Paragon tracks documentation of training for each employee (**Attachment M1-000331-D**). In addition, Paragon submits a sample page from the employee sign-off sheet that demonstrates completion of QA orientation training (**Attachment M1-000331-D**).

Paragon requires every employee to read the LQAP upon hire and annually thereafter, per SOP 143. LQAP review and sign-off is documented through Form 158 and is tracked via the Training Records Tracker spreadsheet. Form 158 follows for your review (**Attachment M1-000331-D**).

Paragon continues to compile the certification files for each analyst. The QA Department has developed a supplemental tracking system in order to better manage SOP review/sign-off and associated IPR demonstration (example template provided as **Attachment M1-000331-D**). In addition, a proficiency program which consists of supervisory sign-off of a job skills checklist, has been instituted to document the competencies of non-analytical personnel (example follows as **Attachment M1-000331-D**).

Corrective Action	Date of Completion	Responsible Parties
Revise Section 14 of LQAP to include more complete definition of training requirements and documentation of training	08/28/00	QA Department
Revise, rename, release SOP 143	08/28/00	QA Department
Training Records Tracker spreadsheet developed and maintained by QA Department	fully instituted; maintenance on-going	QA Department
QA Orientation/Training Sign-off sheet instituted	fully instituted; maintenance on-going	QA Department
Form 158, LQAP Attestation Statement, developed	03/00 initiated; 06/00 fully instituted	QA Department
Supplemental SOP Review/IPR tracking spreadsheet instituted; IPR record compilation initiated	03/00 initiated; IPR completion targeted for 11/30/00	QA Department
Proficiency attestation program instituted for non-analytical	03/00 initiated; completion targeted	QA Department

personnel

for 11/30/00

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Item M1-000331-E: *The PAI instrument calibration program does not have a tag out system for out of calibration equipment. (Priority II) (ICPT BOA, Attachments B and C, Criterion 7).*

Response M1-000331-E: SOP 317, which describes procedures for removing equipment from service and provides guidelines for returning equipment to service, has been revised and released. A copy of SOP 317, Revision 4 and an example of an out-of-service placard follow for your review (*Attachment M1-000331-E*).

Corrective Action	Date of Completion	Responsible Parties
Revise, release SOP 317	09/06/00	QA Department

Data Quality -- Organics

No Findings

Data Quality -- Inorganics

No Findings

Data Quality -- Radiochemistry

Finding M4-000331-A: *Radiochemistry standards are not re-verified annually. (Priority II) (ICPT BOA, Radiochemistry Requirements, Part I, Section 2.9.2)*

Response M4-000331-A: At the time of the audit, Paragon's Radiochemistry Group followed the re-verification guidelines prescribed by SOP 734, Revision 6, which did not require annual re-verification of standards. In order to comply with the ICPT BOA requirement, all radiochemistry standards have been assigned a one-year expiration date from the date of preparation. In addition, SOP 734, Section 5 has been revised to address the ICPT BOA requirement to re-verify standards annually. Revision 7 of SOP 734 follows for your review (*Attachment M4-000331-A*).

Corrective Action	Date of Completion	Responsible Parties
Assign one-year expiration date for all standards	08/16/00	Radiochemistry Operations Manager
Revise, release SOP 734	09/12/00	QA Department

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Finding M4-000331-B: Alpha Spectrometry Instrument logbook entries are not always signed. (Priority II) (ICPT BOA, Special QA Requirements, Criterion 1-7)

Response M4-000331-B: Paragon acknowledges that some logbooks had not been reviewed and signed as a result of oversight. On 08/16/00, all radiochemistry analysts were reminded of the requirement to perform monthly reviews of all logbooks per SOP 328, Revision 1. An internal audit by the QA Department will be conducted on 10/16/00 to ensure compliance with this requirement. Paragon notes that some logbooks do not have routine entries and these will be reviewed upon completion of the page (e.g., maintenance logbooks).

Corrective Action	Date of Completion	Responsible Parties
Radiochemistry Group required to read SOP 328, Revision 1	08/16/00	Radiochemistry Operations Manager
Perform internal audit of logbooks in Radiochemistry Department	10/16/00	QA Department

Finding M4-000331-C: Alpha Spectrometry Calibration curves for energy are generated using only 2 nuclides not the required 3. (Priority II) (ICPT BOA, Radiochemistry Requirements, Part 2, Section 2.2.4).

Response M4-000331-C: Prior to this audit, Paragon's clients have not required that three (3) isotopes be used to generate a calibration curve. Therefore, Paragon has routinely generated curves from two (2) isotopes, Am-241 and U-234. The plated sources used to calibrate the instrument contain three (3) isotopes: Am-241, U-234, and U-235.

As of this writing, Paragon has not successfully calibrated with three (3) isotopes. The peak-fitting routine normally used to process data resulted in a calibration error for the efficiency calibration when three (3) isotopes were used for calibration. Paragon will attempt a region-of-interest (ROI) fitting routine for the three (3) isotopes.

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Corrective Action	Date of Completion	Responsible Parties
Modify calibration practice to include 3 nuclides; calibration via peak-fitting routine; unsuccessful	09/01/00	Radiochemistry Operations Manager
Modify calibration practice to include 3 nuclides; calibration via ROI	10/15/00 (target)	Radiochemistry Operations Manager

Finding M4-000331-D: *The sealed H^3 standard used for daily instrument performance assessment for a Beckman Liquid Scintillation Counter (LSC) expired in 1998. (Priority II) (ICPT BOA, Radiochemistry Requirements, Part I, Section 2.7.2).*

Response M4-000331-D: As a result of oversight, the H^3 source had not been replaced per the manufacturer's expiration date. In order to comply with the requirements of the ICPT BOA, Paragon has replaced the H^3 standard. The H^3 daily check source has been replaced with Beckman #HJS0508, Lot S910156, a 107200 dpm H^3 source that expires on 10/15/04. Attached for your review please find Beckman's documentation for this standard (*Attachment M4-000331-D*).

Corrective Action	Date of Completion	Responsible Parties
Replace H^3 daily check source	08/16/00	Radiochemistry Operations Manager

Finding M4-000331-E: *Instrumentation used for radiological pre-screening analysis is calibrated for attenuation with sources prepared in ringed planchets while samples are prepared in flat planchets. (Priority II) (ANSI N42.25-1997, Annex A).*

Response M4-000331-E: In order to comply with the requirements of the LQAP, Section 7, and the ICPT BOA, Paragon has changed its prescreening practice. As of this writing, all pre-screen samples and calibration sources are prepared in ringed planchets.

Corrective Action	Date of Completion	Responsible Parties
Modify pre-screen practice to ensure that standards and samples are measured in containers having the same geometries	08/16/00	Radiochemistry Operations Manager

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Laboratory Information Management / Electronic Data Management

Finding M5-000331-A: PAI SOPs for the Laboratory Management System (LIMS) do not contain all information for performing the required activities. (Priority II) (ISO 17025, Section 5.4.4 & 5.4.5).

Response M5-000331-A: Paragon has revised the three (3) SOPs to reflect the current state of our LIMS. Please note that the SOPs have also been renumbered. The SOPs follow for your review (*Attachment M5-000331-A*).

Corrective Action	Date of Completion	Responsible Parties
Revise, renumber SOPs for LIMS procedures	09/01/00	QA Department, IS Department

Hazardous and Radioactive Materials Management

Note: Paragon has retained the services of Montgomery & Associates of Idaho Falls, Idaho to assist us in addressing waste management and radiation safety concerns. Mr. Robert Montgomery, Principal, performed an on-site audit of Paragon on April 24-27, 2000. Following his audit, Mr. Montgomery has been preparing documents per the suggestions of the ICPT auditor and federal regulations. Mr. Montgomery will return to Paragon in November 2000 for one week to provide on-site training on waste management and radiation safety issues to all employees.

Finding M6-000331-A: PAI does not have a formally documented Radiation Protection Program. (Priority II) (10CFR20.1101).

Response M6-000331-A: Paragon acknowledges that some elements of the Radiation Protection Program are incomplete and that the elements should be integrated. Montgomery & Associates has been hired to rewrite and create linkages among several documents, including the: radiation safety manual; radiation safety SOPs; and waste management plan. Paragon will provide copies of the final documents upon request. Paragon anticipates that these documents will be finalized by 11/30/00.

Paragon's LIMS has been programmed to update the radionuclide inventory (based on pre-screen data), which enables us to manage any H&S concerns related to particular samples and to maintain an accurate inventory of radionuclides. This module of LIMS has been functional since July 2000.

Additional elements of the Radiation Protection Program have been functional for several years. For example, the personal dosimetry program requires quarterly monitoring of all

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laboratory employees. In addition, weekly swipes are taken throughout the laboratory in order to monitor removable radiation and monthly external radiation dose surveys are also performed.

Corrective Action	Date of Completion	Responsible Parties
Revise radiation safety manual, radiation safety SOPs, and waste management plan	11/30/00	H&S Department
Program LIMS to update radionuclide inventory	~07/00, fully functional LIMS module	IS Department

Finding M6-000331-B: *The contents of the PAI RCRA Contingency Plan are inadequate. (Priority II) (6 CCR 265, Subpart D).*

Response M6-000331-B: Montgomery & Associates is rewriting the RCRA Contingency Plan per 6 CCR 265, Subpart D and CDPHE guidance. Following Paragon's approval of the document, Mr. Montgomery will provide on-site training for all employees. Paragon will provide copies of the final document upon request.

Corrective Action	Date of Completion	Responsible Parties
Rewrite RCRA Contingency Plan	09/30/00	H&S Department, QA Department
Provide on-site training for all employees	11/30/00	Montgomery & Associates

Finding M6-000331-C: *The contents of the PAI Chemical Hygiene Plan are inadequate. (Priority II) (ICPT BOA, Attachment 1, Section 2.2.8).*

Response M6-000331-C: Montgomery & Associates is rewriting the Chemical Hygiene Plan (CHP). Following Paragon's approval of the document, Mr. Montgomery will provide on-site training for all employees. The revised CHP will include the RCRA Contingency Plan and links to the H&S SOPs and radiation protection plan. Paragon will provide copies of the final document upon request.

Corrective Action	Date of Completion	Responsible Parties
Rewrite Chemical Hygiene Plan	09/30/00	H&S Department, QA Department
Provide on-site training for all employees	11/30/00	Montgomery & Associates

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Finding M6-000331-D: *The operation of the PAI wastewater treatment unit is not adequately described or formally controlled. (Priority II) (ICPT BOA, Attachments B and C, Special Laboratory Requirements, Section 4).*

Response M6-000331-D: In order to address this finding, Paragon will revise SOP 017, which addresses the operation of the wastewater treatment unit. In addition, the Waste Management Plan that is being revised by Montgomery & Associates will address operational requirements as required by the Boxelder Sanitation District.

Any sludge generated by the wastewater treatment unit will be managed as a hazardous waste. Paragon will perform required organic and inorganic analyses in order to create an accurate profile of the sludge and to ensure that any constituents present in concentrations greater than the underlying hazardous constituents level are listed on the land disposal restriction form.

Corrective Action	Date of Completion	Responsible Parties
Rewrite Waste Management Plan	09/30/00	H&S Department, QA Department
Provide on-site training for all employees	11/30/00	Montgomery & Associates
Revise SOP 017	11/30/00	H&S Department, QA Department

Finding M6-000331-D: *Potentially radioactive sample processing waste is not segregated from sanitary trash.*

Response M6-000331-D: In order to address this finding, Paragon developed a contact waste collection system that was implemented by 04/15/00. During Mr. Montgomery's audit of May 2000, he reviewed all SAAs and trash receptacles to ensure that Paragon's segregation practices and labeling were compliant. Therefore, we believe that our radioactive sample processing waste is appropriately segregated from sanitary trash. The revised Waste Management Plan and SOP 003 will address characterization protocol for contact waste. Copies of these documents are available upon request.

Corrective Action	Date of Completion	Responsible Parties
Develop contact waste collection system	04/15/00	H&S Department
Rewrite Waste Management Plan	11/30/00	Montgomery & Associates, H&S Department, QA Department
Revise SOP 003	10/30/00	H&S Department
Provide on-site training for all employees	11/30/00	Montgomery & Associates

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Finding M6-000331-F: PCB wastes are not managed in compliance with the Toxic Substances Control Act (TSCA). (Priority II) (ICPT BOA, Attachments B and C, Special Laboratory Requirements, Section 4).

Response M6-000331-F: Following the audit, Paragon labeled all areas in which PCBs were stored. Paragon has labeled the PCB containers with the EPA-mandated label: "*Caution Contains Polychlorinated Biphenyls.*"

Mr. Montgomery verified labeling practices during his audit in April 2000.

The revised Waste Management Plan will address PCB waste management per TSCA regulations.

Corrective Action	Date of Completion	Responsible Parties
Label all areas in which PCBs are stored	04/15/00	H&S Department
Revise Waste Management Plan	11/30/00	Montgomery & Associates, H&S Department, QA Department

Finding M6-000331-G: The PAI waste management plan does not reflect current practices and is not adequate in describing many ongoing waste processing activities.

Response M6-000331-G: Montgomery & Associates is rewriting the Waste Management Plan in order to address this finding.

Corrective Action	Date of Completion	Responsible Parties
Revise Waste Management Plan	11/30/00	Montgomery & Associates, H&S Department, QA Department

Finding M6-000331-H: The process for disposition of the samples that have exceeded their archival date is not adequately documented or implemented.

Response M6-000331-H: Paragon has developed a module in our LIMS system that tracks samples through their archival period. This module allows us to identify samples that are characterized and ready for disposal. LIMS generates batch reports for samples ready for disposal and segregates the samples into appropriate waste

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streams. Attached for your review please find a sample of a LIMS report (*Attachment M6-000331-H*).

In addition, Paragon has dedicated additional resources to sample archiving and disposal. Ms. Becky Wasson has been assigned the responsibility of managing the sample archiving and disposal process and is assisted by technicians from each group.

Finding M6-000331-I: PAI is not performing biennial reviews of waste profiles as required.

Response M67-000331-I: Paragon's revised Waste Management Plan will address the requirement of performing biennial reviews in order to verify our waste profiles. In addition, the Sampling and Analysis Plan will be written to comply with 40CFR 265.13 (a&b) and 6CCR1003-7, 265.13 (a&b).

Corrective Action	Date of Completion	Responsible Parties
Revise Sampling and Analysis Plan	11/30/00	H&S Department
Revise Waste Management Plan	11/30/00	Montgomery & Associates, H&S Department, QA Department

Item M6-000331-J: *The process for identifying incoming samples that require a prescreen for radioactivity analysis is informal. (Priority II) (ICPT BOA, Attachments B and C, Special Laboratory Requirements, Section 5)..*

Response M6-000331-J: Paragon believes that our process for identifying incoming samples that require a prescreen for radioactivity is well defined and thoroughly documented. Paragon's Project Managers work with clients to define all technical and service requirements prior to receipt of samples. This interview includes questions about potential radioactivity (e.g., site history, historical data, expected radionuclides and levels of activity). Project Managers distill project requirements to all Sample Receipt and Operations personnel by issuing Program Specifications. This information is generated through the LIMS and addresses health and safety and waste disposal information -- including prescreen requirements. The Sample Receiving staff determines which sites/samples require prescreen from this information. A sample Program Specification follows for your review (*Attachment M6-000331-J*).

In the event that samples arrive unannounced, the Sample Receiving staff place the samples on "hold" status and forward Chain of Custody information to the Operations Manager. The Radiation Safety Officer and Operations Manager assess the new client's prescreen requirements via a teleconference with the client. These requirements are then conveyed to Sample Receipt and Operation personnel via Program Specifications.

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In general, Paragon performs a prescreen for radioactivity on all samples received from DOE sites or on behalf of a DOE site, unless the client provides reliable prescreen data.

Paragon has maintained a database of radioactive samples (based on prescreen data) and standards since 1993, which enables us to monitor our inventory of radioactive materials. Samples pages of the historical database from 1993-1994 follow for your review (*Attachment M6-000331-J*). In April 2000, 5247 records from the historical database were transferred to the LIMS as .dbf files. Paragon's LIMS automatically links client, project, work order, volume, and radionuclides in the module that manages radionuclide inventory. Samples pages from the LIMS database follow for your review (*Attachment M6-000331-J*).

Corrective Action	Completion Date	Responsible Party
Create database of radioactive samples and standards in order to monitor inventory of radioactive materials	1993	H&S Department
Transfer historical database to LIMS for continued management of radioactive materials	04/2000	H&S Department, IS Department

Item M6-000331-K: *Radioactive sample shipments and potentially radioactive samples are not surveyed for internal surface contamination before sample handling. (Priority II) (PAI CAP response to a DOE Oak Ridge audit).*

Response M6-000331-K: PAI will institute a removable contamination survey program for sample containers that contain radioactive material shipments. The types of shipments that will undergo sample container removable radioactive material contamination surveys include: excepted radioactive material packages; low specific activity packages; radioactive I, II, or III packages; and any shipment from a client that has potential radioactive contamination.

The most common types of packages to be received at Paragon are excepted radioactive material and radioactive I packages. The sample containers will be subjected to a composite removable radioactive material contamination survey (swipe). The swipe will be counted for 5 minutes by both the Ludlum 1000 Scaler with 43-10 Alpha Scintillation Detector for detection of alpha particles and the Ludlum 1000 Scaler with 44-7 Geiger-Mueller Detector for detection of Beta/Gamma emissions. Action levels are 20 dpm/100 cm² removable alpha and 200 dpm/100 cm² removable beta/gamma (Nuclear Regulatory Commission's Decommissioning Release Limits for Unrestricted Use).

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If the composite sample's results are less than the above limits, the samples will be released. If the limits are exceeded on the composite swipe, then all containers must be swiped individually in order to locate the source of radioactivity. The Sample Receiving staff will be trained to perform composite removable radioactive material swipes on sample containers and to evaluate results on 09-18-00.

Form 009, a worksheet that calculates and presents swipe sample values, has been prepared. SOP 008 has been revised. Copies of these documents follow for your review (*Attachment M6-000331-K*).

Sample Receiving staff will be trained to perform swipes on 09-18-00. Paragon will survey shipments for internal surface contamination as of 09-19-00.

Corrective Actions	Date of Completion	Responsible Parties
Prepare incoming sample removable radioactive material contamination survey log form (Form 009)	09-10-00	H&S Department
Revise SOP 208 to include incoming sample removable radioactive material contamination survey	09-14-00	H&S Department, QA Department
Train sample receiving staff to prepare incoming sample removable radioactive material contamination survey	09-18-00	H&S Department
Begin surveying shipments for internal surface contamination	09-19-00	Sample Receiving Staff, H&S Department

Observations

Quality Assurance Management Systems

Item 1: *The response time for corrective action response to audit findings is currently unacceptable. The response to the Oak Ridge audit of December 7, 1999 was not completed until March 25, 2000. The response for the July audit by INEEL was not received until December 1999. IT is expected that PAI will respond within 30 days from receipt of the final EMCAP audit report.*

Response 1: Paragon apologizes for delays in submitting written responses. We will strive to complete responses in a more timely fashion.

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Data Quality -- Organics

Item 1: *An unlabeled sample container was located in the TCLP extraction laboratory; however, the chemist immediately corrected the problem by labeling the beaker.*

Item 2: *Florisil lot checks are not being performed on a consistent basis.*

Response 2: Paragon's gc/svoa group has created a logbook in which they file the chromatograms for each Florisil lot's evaluation. Paragon evaluates each lot of Florisil per the criteria in the CLP SOW, Section D-54/PEST, Section 10.1.8.2.2.4. Sample pages from the logbook follow for your review (*Attachment Observations, Organics, Item 2*).

Item 3: *An explosion proof refrigerator is needed in the organic extractions laboratory to store herbicide extracts. At the present time, the extracts are stored in the sample storage refrigerator.*

Response 3: Paragon concurs that an explosion-proof refrigerator is preferable for storing herbicide extracts. When the current refrigerator fails, we will replace it with an explosion-proof one.

Item 4: *A refrigerator is needed in the organic extractions laboratory for the storage of semivolatile extracts.*

Response 4: Paragon stores semivolatile extracts in two (2) dedicated refrigerators. One of these refrigerators is located in the organic extractions laboratory and the other in the gc/ms semivolatiles laboratory.

Data Quality -- Inorganics

Item 1: *The TSS laboratory is not monitored or documented. This was the only drying oven found which (sic) the temperature was not monitored or documented within the inorganic section. The PAI QA department should ensure that all drying ovens are monitored and documented laboratory wide.*

Response 1: The oven in question was not used to perform solids determinations at the time of the audit. This oven was only used to dry glassware and therefore was not monitored daily. All drying ovens for which temperatures are

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prescribed are monitored daily. Pages from the three (3) inorganics drying ovens follow for your review (*Attachment Observations, Inorganics, Item 1*).

Item 2: *Some maintenance logbooks in the inorganic area were missing the instrument identification and its serial number.*

Response 2: Following the audit, the inorganics laboratory was instructed to include instrument identification and serial numbers in all logbooks. The QA Department will perform a laboratory-wide audit of logbooks in October to ensure that logbooks are properly documented and reviewed, per our SOPs.

Item 3: *The storage cooler in the organic laboratory does not have a contingency plan for refrigerator failures to protect sample integrity in case of temperature failure. It is recommended that PAI institute as a (sic) contingency plan in case of cooler temperature failure. South Carolina certification requires that refrigerators be monitored twice a day. One refrigerator was monitored once a day.*

Response 3: PAI has approximately 12 refrigeration units throughout the laboratory that may be used as "temporary" storage areas in case of failure. Movement of samples -- as a result of failure -- is documented in logbooks (e.g., RU-20, Sample Control, 08/18/00).

The QA Department has reminded all groups that refrigerators shall be monitored twice daily, Monday through Friday. The laboratory depends upon wheel-chart recorders during the weekends. These wheel-chart recorders are verified quarterly.

The QA Department will perform a laboratory-wide audit of refrigeration units in October to ensure that they are monitored twice daily and that logbooks are properly documented and reviewed, per our SOPs.

Item 4: *Monitoring of the metals digestion water bath temperature need improvement. PAI should evaluate the temperature variation in metals water bath to ensure that it is following closely the temperature required by the meals digestion SOP.*

Response 4: Paragon believes that this observation may be the result of a misunderstanding, as the metals digestion water bath temperature was and is monitored by a thermometer. Pages from the time of the audit, March 30-31, demonstrate compliance and follow for your review (*Attachment Observations, Inorganics, Item 4*). Inspection of the current logbook reveals that the water temperature has been measured at 93-95 °C, which meets the requirements of Method SW3005A.

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Data Quality -- Radiochemistry

Item 1: *The batch QC is listed first on the batch worksheet for radiochemistry. For this reason, the blank and LCS appear to be in the same position in the Gas Proportional counter (GPC), and same sample preparation apparatus (i.e., tritium distillation).*

Response 1: As of 08/16/00, the radiochemistry laboratory began listing batch QC samples at the end of the bench sheet.

Laboratory Information Management / Electronic Data Deliverables

None

Hazardous and Radioactive Materials Management

Item 1: *A document hierarchy was not readily apparent at PAI. For example, it was difficult to determine how the regulatory requirements were incorporated in facility plans and then implemented in the SOPs. The plans did not cross-reference each other or contain links to the appropriate SOPs.*

Response 1: As stated above, Paragon has retained the services of Montgomery & Associates of Idaho Falls, Idaho to assist us in addressing waste management and radiation safety concerns. Mr. Robert Montgomery, Principal, performed an on-site audit of Paragon on April 24-27, 2000. Following his audit, Mr. Montgomery has been preparing documents per the suggestions of the ICPT auditor and federal regulations. Mr. Montgomery is aware that linkages need to be created among documents.

Item 2: In the organic extractions laboratory, radiation trefoil stickers were observed in the sanitary trash and the container was labeled for broken glassware.

Response 2: Paragon developed a contact waste collection system that was implemented by 04/15/00. During Mr. Montgomery's audit of May 2000, he reviewed all SAAs and trash receptacles to ensure that Paragon's segregation practices and labeling were compliant.

Item 3: *In the organic extractions laboratory, broken condensers were being used.*

Response 3: Paragon has disposed of all broken glassware in the organic extractions laboratory.

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Item 4: *In the organic extractions laboratory, the roof was leaking. This could possibly contaminate samples.*

Response 4: Paragon has repaired the leaking roof in the organic extractions laboratory.

Item 5: *In the organic extractions laboratory, PAI personnel were not wearing prescription safety glasses.*

Response 5: Paragon has provided vouchers for prescription safety glasses to all employees (as needed).

Item 6: *In the organic extractions laboratory, a non-tapped wire was protruding from an inside wall near the building exit at the GPC area.*

Response 6: Paragon has removed the wiring in question.

Item 7: *A program for periodic chemical exposure monitoring has not been defined.*

Response 7: The H&S Department is developing a chemical exposure monitoring plan. As of this writing, monitoring of the VOA laboratory has begun.

Thank you again for your time and assistance during the on-site audit. We hope that our responses meet your requirements. Please contact me at 970 490 1511 if additional information is required and I will be glad to provide it.

Respectfully Submitted,

Debra Henderer
Quality Assurance Manager
Paragon Analytics, Inc.

Enclosures



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Ms Debra Henderer
Laboratory QA Manager
Paragon Analytics, Inc.
225 Commerce Drive
Ft. Collins, CO 80542

August 18, 2000

Laboratory Quality Audit

Enclosed is the observation report for the on-site analytical laboratory audit at Paragon Analytics, Inc. in Fort Collins, CO, performed on 8/16 - 8/17/00, by IT Corp. It is the opinion of this auditor that the quality program and analytical systems used throughout the laboratory are adequate for Paragon Analytics to provide analytical services to IT Corp. in support of the Rickenbacker Air Force Base (RANGB) Delivery Order (DO) 19.

There were no findings during the audit, but two observations have been noted. There are no required responses for the observations noted.

I appreciated the opportunity to work with you and your organization, and the cooperation and cordiality afforded me during the audit was refreshing. I apologize for any inconvenience I may have caused by my interruptions. I look forward to working with you on the upcoming projects.

If there are any questions, please feel free to call me at (513) 782-4699 at any time.

Respectfully,

A handwritten signature in cursive script that reads 'Bruce H. Rohrbach' followed by the date '9/12/00'.

Bruce H. Rohrbach
Senior QA/QC Chemist
PAWMS North Area Technical Lead

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The meeting was initiated on Wednesday morning August 16th at 8:30 AM with the following in attendance:

Don Gipple - Laboratory Director - Paragon

Lori Pacheco - Operations Manager - Paragon

Debra Henderer - Quality Assurance Manager - Paragon

Peter Gintautas - Technical Manager - Paragon

Bruce H. Rohrbach - Senior QA/QC Chemist - IT Corp.

The reason for the audit was stated and general discussion of what was expected and the outline of activities were presented. A tour of the facility prior to the audit followed and the audit was initiated prior to breaking for lunch.

Each major laboratory functional area of concern was reviewed and discussions regarding the instrument standardization, tune and calibration were performed. All items were determined to be acceptable, unless otherwise indicated.

Attached to the end of this report are examples of the laboratory's checklists used to assure the quality of the data generated and reviewed.

Paragon Analytics, Inc.
Laboratory Audit 8/16/00 & 8/17/00

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Audited Area	Comments	Observations
Sample Receiving (Mark Stella)	Basically a one-man operation. Sample transports are screened with Geiger counter to determine presence of radioactive species. Samples are all checked for temperature with infrared detector. Manually assigned a workorder number, which stays with samples throughout processes in lab. Distributes samples to actual lab for storage. Prepares samples kits for clients.	Sample receiving clerk not in proper PPE at all times. Initially without lab coat and eventually working without protective gloves while handling the sample containers. Issue was discussed during audit and closed.
Laboratory Information Management System (LIMS)	Workorder entered into in-house LIMS (based upon Microsoft Access Database). Flexible system maintained by staff member.	None. LIMS is work-in-progress, but an excellent tool as reviewed.
Sample Control (Cheri Matha)	Continues the log-in process and supplies information regarding specific client tests. Paperwork finished and transferred to PM to review. Finishes and corrects all input.	None
Sample Storage	Samples are stored within operational area of laboratory, not in a main repository. Samples logged out by section analysts.	None
Volatile Mass Spectrometry (Tyler Knaebel)	Three HP instruments available for analysis, plus 1 Arcon auto-sampler. Service contract provided by Full Spectrum. In process of training new employee transferring from another area. Reviewed and discussed instrument tune and calibration procedures determined to be acceptable. Reviewed checklists used by analysts during data review. Good format.	None Room felt warm, but apparently stable for instruments.

Paragon Analytics, Inc.
Laboratory Audit 8/16/00 & 8/17/00

Audited Area	Comments	Observations
Organic Compound Extractions (Eric Bayless)	Adequate area available for performance of functions. Primarily perform continuous liquid extractions and soxhlet extractions for solids. Can perform separatory funnel extractions for liquid and sonication for solids. Procedures acceptable and in order. Use kiln to dry glassware following washing, which they say help the cleanliness.	None
Semivolatile Mass Spectrometry (Marty Brown)	Three HP instruments available for analysis along with two analysts and a trainee analyst. Additional individual to compile data for packages. Reviewed and discussed instrument tune and calibration procedures determined to be acceptable. Excessive sample backlog in this area and spent less time here in order not to affect lab throughput.	None Amount of review applied to data seems somewhat excessive, but provides clean data and little problems for the client
Metals; Analysis and Preparation (Darryl Patrick)	Five-man operation handling entire process from preparation through analysts to reporting. Sample preparation somewhat unique: samples are diluted to final weight rather than final volume (i.e. sample is weighed constantly and the final volume of water is weighed in). Soil samples are not filtered following preparation (allow to settle out). Mercury preparation uses more sample weight than recommended in the present SW-846 method. However, is consistent with update to procedure now pending. Reviewed and discussed instrument standardization and calibration procedures determined to be acceptable.	None

Paragon Analytics, Inc.
Laboratory Audit 8/16/00 & 8/17/00

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Audited Area	Comments	Observations
Standard Operating Procedures (SOPs)	Specific SOPs were reviewed and additional ones requested for later review, which have not yet been received. Code of Ethics Documentation Laboratory Quality Assurance Plan (LQAP) review policy. Ethics and Data Integrity. Requested and received list of pertinent laboratory SOPs. Review is in process, but expect no surprises.	Various documents still retained header information of ATI, the previous name of the facility. Process to changeover is long and laborious.
Training Records	Reviewed the Hazardous Waste Management Employee Training.	None
Employee Performance Records	Reviewed the entire file for three randomly chosen employees. The records contain resumes, transcripts, and performance results of samples prepared and analyzed.	None
QA Files	Reviewed the results of both Internal and External Audits. Reviewed the thermometer temperature calibration. Reviewed balance and weight calibration. Reviewed the non-conformance reports (NCRs) file.	None
Laboratory Quality Assurance Plan (LQAP)	LQAP was reviewed initially 6 months ago during request for proposal (RFP) process for another project. Review indicated document received during this trip is identical to previously reviewed document.	None
Sample Disposal	Area within lab designated as the collection repository for all finished sample material. Analysis referred to determined the degree of hazard of the material and segregated as such.	None



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225 Commerce Drive ♦ Fort Collins, CO 80524 ♦ (800) 443-1511 ♦ (970) 490-1511 ♦ FAX (970) 490-1522

Via Federal Express

March 25, 2000

Mr. David Carden
Laboratory Audit Program Manager
Waste Management and Technical Integration Team
Environmental Management (EM-921)
U.S. Department of Energy
P.O. Box 2001
Oak Ridge, TN 37830

**Re: Department of Energy Oak Ridge Operations
Laboratory Qualification Audit of Paragon Analytics, Inc.
December 6-7, 1999
Paragon's Corrective Action Report**

Dear Mr. Carden:

I am writing to respond to Mr. Gist's report of January 27, 2000. Paragon sincerely appreciates the DOE-ORO on-site audit of our systems and processes and the time spent with our employees. We are pleased to respond to the 20 findings and 16 observations from the audit. Paragon's responses and corrective actions follow for your review.

Findings

Quality Assurance Management Systems

Item QA-991207-A: PAI is not effectively performing periodic reviews and updates on their Standard Operating Procedures (SOPs). (Priority II) (DOE Order 414.1)

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Response QA-991207-A: Paragon acknowledges that we have not completed the review and revision of all SOPs within the past year. As of this writing, 176 SOPs have been reviewed and revised in the past year. Approximately 45 SOPs have not yet been reviewed and revised in the past year. These 45 SOPs will be reviewed and revised by April 30, 2000. Attached for your review please find a table of contents for SOPs that demonstrates the latest date of review and revision (*Attachment QA-991207-A*).

Corrective Action	Date of Completion	Responsible Parties
176 SOP updates completed	03/17/00	QA Department
45 SOP updates to be completed	04/30/00	QA Department

Item QA-991207-B: *Logbooks are not being reviewed on a consistent basis. (Priority II) (SOP 328, Review of Logbooks)*

Response QA-991207-B: Paragon acknowledges that review of laboratory logbooks was not being conducted per the monthly time frame stated in SOP 328 at the time of the audit. This finding and directives for corrective action were communicated to all laboratories after the audit. Follow-up reviews to ensure documented review and a labwide (refresher) training were conducted on 03/13/00. An internal audit of laboratory logbook review is scheduled for 08/16/00. Attached for your review please find documentation of the labwide training, copies of SOPs 303 and 328, and the internal audit schedule (*Attachment QA-991207-B*).

Corrective Action	Date of Completion	Responsible Parties
Verbal reminder of laboratory logbook review issued to Department Managers	12/01/99	QA Department
Informal audit to ensure documented laboratory logbook review	03/13/00	QA Department
Labwide training (logbook review) conducted re: SOPs 303 and 328	03/13/00	QA Department
Formal internal audit (logbook review) scheduled	08/16/00	QA Department

Item QA-991207-C: *Statistical control charts are not maintained in real time and are not monitored on a routine for the analysis of trends and biases. (Priority II) (SW-846 Chapter One, Section 4.4.2)*

Response QA-991207-C: Every 6 months, the IS Department provides statistical control charts to the QA Department for all LCS/LCSD data points in our LIMS database.

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These control charts include the following information: analytical method; extraction method; matrix; number of observations (n); minimum recovery; maximum recovery; standard deviation ($\pm\sigma$); warning limits ($\pm 2\sigma$); control limits ($\pm 3\sigma$); 2-dimensional plot (percent recovery vs. date/observation). The QA Department reviews the data for every method and matrix and updates qc limits if necessary (update is usually an annual one).

Prior to the DOE ORO audit, the approach described above had been accepted by all auditors and clients. Per the DOE ORO auditor's request, Paragon will program two (2) statistical outlier tests in order to monitor control charts for trends and biases and use this information to monitor performance. Paragon's IS Department is programming a Grubbs Test and Dixon Outlier Test. We anticipate that programming and testing will be completed by April 30, 2000.

Corrective Action	Date of Completion	Responsible Parties
Program and evaluate Grubbs and Dixon Outlier Test in LIMS	04/30/00	QA and IS Departments

Item QA-991207-D: *PAI definition of training requirements and record maintenance are not adequate (Priority II) (DOE Order 414.1; Quality Assurance; 40 CFR Part 262).*

Response QA-991207-D: Paragon acknowledges that our training program and documentation of training requires development and maintenance. We have begun a comprehensive training program for all aspects of laboratory operations. This training program will include training modules for: human resources, quality assurance, health and safety, general lab operations, and departmental operations. These programs will define the required training for each staff member.

To comply with the concerns of this finding the following actions are being taken: (1) The current training records are being entered into the training database. (2) The Chemical Hygiene Plan and applicable SOPs will be revised to include the training requirements for RCRA Waste Management Staff. (3) The new Health and Safety training matrix will include RCRA Waste Management Training Requirements. (4) The training and retraining requirements for Radiation Safety and Chemical Hygiene for both analytical and nonanalytical workers will be placed in the Radiation Safety Manual and Chemical Hygiene Plan as applicable. The retraining requirements will also be defined in the new Training Matrices.

Corrective Action	Date of Completion	Responsible Parties
Input existing training records into health and safety training database	05-15-00	H&S Department
Complete health and safety training matrices	04-05-00	H&S Department

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Revise Chemical Hygiene Plan to include health and safety and RCRA waste management training requirements	05-15-00	H&S Department
Define retraining requirements and frequency for health and safety, radiation safety, and RCRA waste management for all workers	03-20-00	H&S Department



Sample Control and Laboratory Information Management Systems (LIMS)

Item SC-991207-A: The temporary storage refrigerator in sample receiving does not use refrigerator blanks to monitor for volatile cross contamination. (Priority II) (Analytical Master Specification (AMS) Appendix D Attachment J-II, section 8.2)

Response SC-991207-A: Paragon acknowledges that at the time of the audit the Sample Control walk-in cooler (RU #20), which is used for temporary storage of samples, was not included in the refrigerator blank monitoring program. Prior to the DOE ORO audit, Paragon analyzed refrigerator blanks on a *weekly* schedule for the GC/MS and GC Volatiles laboratory per the requirements of SOP 512. Per the auditor's request, Paragon now prepares and analyzes a refrigerator blank for the Sample Control area in order to monitor volatile cross contamination in this temporary storage area. The GC/MS Volatiles Group is responsible for preparing, analyzing, and documenting the Sample Control area refrigerator blanks. SOP 512 provides for the weekly analysis of refrigerator storage blanks and has been revised to include refrigerator blank preparation and analysis for RU #20. Attached for your review please find revised SOP 512, Revision 5 and examples of documentation of RU #20 weekly refrigerator blank analysis (*Attachment SC-991207-A*).

Corrective Action	Date of Completion	Responsible Parties
SOP 512 revised to include Sample Control RU #20 in refrigerator monitoring blank program	01/10/00	QA Department
Inception of RU #20 refrigerator blank analyses	01/13/00	GC/MS Volatiles Group

Data Quality -- Radiochemistry

Item DR-991207-A: SOPs for radiological prescreening analysis do not consistently reflect the practice employed by the laboratory analysts. (Priority II) (DOE Order 414.1).

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Response DR-991207-A: Paragon acknowledges that SOPs 703 and 705 required revision at the time of the audit. These SOPs were recently revised in order to reflect current practices. Attached for your review please find revised SOPs 703 and 705 (*Attachment DR-991207-A*). Paragon will recalibrate the screening instrument (LB5100) by 04/05/00.

Corrective Action	Date of Completion	Responsible Parties
Revise SOP 703 to reflect current practices	03-17-00	QA Department, Radiochemistry Department
Revise SOP 705 to reflect current practices	03-16-00	QA Department, Radiochemistry Department
Recalibrate LB5100	04-05-00	Radiochemistry Department

Data Quality -- Inorganics

Item DI-991207-A: *Mercury samples are not prepared for triplicate analysis according to Method 7471. (Priority II) (SW-846, Method 7471).*

Response DI-991207-A: Paragon understands that the intent of multiple measurements is to ensure that the laboratory analyzes a representative sample aliquot. Therefore, we follow US Army Corps of Engineers guidance and SW-846 Method 7471B guidance and weigh out ~0.6 g for a single analysis (instead of ~0.2 g for a triplicate analysis). Paragon discloses this approach in SOP 812, Revision 6, Sections 8.2.2 and 10.1. Paragon respectfully requests your consideration of this equivalent approach.

Data Quality -- Organics

Item DO-991207-A: *Volatile Organic Standards are being stored in the freezer section of the refrigerator used to store unanalyzed samples. (Priority II) (Oak Ridge Site Specific Terms and Conditions, Appendix D).*

Response DO-991207-A: Paragon acknowledges that storage of volatiles standards and samples in the same unit may result in cross contamination. In order to correct the storage conditions, Paragon purchased a stand-alone freezer for the separate, dedicated storage of volatiles standards in December 1999. On 01/10/00, the QA Department verified that all volatiles standards had been segregated from samples and placed in the

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new freezer. Attached for your review please find logbook pages from the new freezer, RU #29 (*Attachment DO-991207-A*).

Corrective Action	Date of Completion	Responsible Parties
Purchase stand-alone freezer for volatiles standards	12/99	GC/MS Volatiles Group
Move volatiles standards to freezer and begin daily monitoring of freezer's temperature	01/05/00	GC/MS Volatiles Group
Verify that all volatiles standards are segregated from samples and stored in new freezer	01/10/00	QA Department


Item DO-991207-B: Documentation and preparation for the TCLP extraction is inadequate. (Priority II) (SW-846, Method 1311 and SOP 609).

Response DO-991207-B: Forms 623 and 608 (bound into logbooks) are used to record TCLP preparation. These forms have been revised to include filtration date and time, initial and final room temperature, and particle-size reduction. Form 646 has been created to address the requirement to record preparation of the TCLP preparation fluids. Attached for your review please find Forms 623, 608, and 646 (*Attachment DO-991207-B*).

As noted by the auditors, Paragon uses HDPE Nalgene containers for leaching organic analytes, instead of borosilicate glass jars as described in SW-846 1311. Paragon's historical data for method blanks and laboratory control samples do not contain contaminants above the reporting limit. Therefore, Paragon believes that the substitution of HDPE Nalgene containers is an acceptable practice. Paragon has revised SOP 609 to include disclosure of this container change in Section 9. Attached for your review please find SOP 609 (*Attachment DO-991207-B*).

One of the auditor's comments in the discussion section of this finding indicated that particle size reduction is not being performed for solid samples. Paragon *does* perform particle size *evaluation* for all solid TCLP samples. If, based upon visual inspection, particle size is deemed to be greater than 0.5%, then the solid sample is passed through a sieve. Form 623 has been revised to provide better documentation of particle size evaluation/reduction. These practices have also been clarified in the revision of SOP 609.

Corrective Action	Date of Completion	Responsible Parties
TCLP extraction Forms 623 and 608 updated to include all required information	01/13/00	QA Department, Organic Extractions Group
Form 646 created to document the preparation of TCLP extraction	03/13/00	QA Department, Organic Extractions

fluids		Group	<div>CONFIDENTIAL</div> <div>DO NOT COPY</div> 
TCLP extraction SOP 609 revised to document use of HDPE Nalgene containers and clarify particle size evaluation/reduction	03/17/00	QA Department Organic Extractions Group	

Materials Management

Item SH-991207-A: *Housekeeping is inadequate in several laboratory areas. (Priority II) (PAI Chemical Hygiene Plan).*

Response SH-991207-A: Paragon acknowledges that the laboratory was in need of general housekeeping at the time of the audit. Since the audit, the laboratory areas have been cleaned and reorganized. Items of note are as follows:

- (1) The auditor noted that boxes and equipment were stored in the hallways. Boxes have been removed from the hallways. New equipment has been removed from the hallways and installed. An older Radioactive Material Hand and Foot Monitor was donated to Colorado State University. In addition, a 16 ft by 25 ft (400 ft²) storage room was constructed in the radiochemistry laboratory common area, which has alleviated clutter. This room has five 8 ft x 10 ft x 4 ft shelving units. This increase storage area has allowed PAI to remove all shelving from the radiochemistry common area and hallways with exception of one staging area for the radiochemistry instrument lab. Currently, the only samples stored in this hallway area are those staged for analyses in the radiochemistry instrument lab. The only other items that remain in the hallways are the Hand and Foot Monitors and safety equipment.
- (2) The auditor noted that containers of waste were staged in laboratory areas. It is true that Paragon uses Satellite Accumulation Areas (SAA) for initial waste collection before moving waste to the 90-day accumulation area. We understand that the auditor would have preferred the laboratory to use one 55-gallon container for each kind of waste. Instead, Paragon's practice is to use multiple, smaller (5-gallon) containers so that we may transfer waste into the 5-gallon carboys inside the fume hoods. (The 5-gallon carboy is the largest size that can be moved into the fume hoods.) The State of Colorado Department of Public Health and Environment has advised us that the use of multiple containers for one kind of waste stream in a SAA is allowable, with a maximum of 55 gallons of any one waste stream in the SAA. To ensure compliance with the 55-gallon limit, Paragon allows a maximum of ten 5-gallon carboys of each waste stream to be present in each SAA.

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- (3) The auditor noted dusty work areas. Paragon has addressed this finding by assigning daily cleaning responsibilities to every analyst in each laboratory. Analysts are responsible for ensuring that fume hoods and countertops are cleaned daily. In addition, Paragon has increased the frequency and responsibilities of the off-hours cleaning crew. The cleaning crew cleans the facility five (5) days per week (Monday through Friday). The cleaning crew's general duties for common areas and laboratory areas include: sweeping and wet mopping floors, dusting, removing sanitary trash, and discarding empty boxes.
- (4) The auditor noted that trash and debris were present on the Sample Receiving Area floor. This area is a high-traffic one in which many operations are performed that generate trash and debris throughout the work day (e.g., packing materials, boxes). As a result, this area is prone to become dirty and cluttered throughout the work day. Paragon has taken the following steps to alleviate the problem: (1) The after-hours cleaning regime has been increased to include sweeping every day and wet mopping three times weekly. (2) The sample receiving staff has been assigned the responsibility of cleaning the area during sample unloading and supply unpacking operations (throughout the day).
- (5) The auditor noted that some laboratory hoods were crowded with waste containers, sample containers, and chemical reagents. The areas affected were the waste characterization and organic extractions laboratories. The waste and sample containers in these laboratories have been removed. The hoods in the waste characterization laboratory have been cleared of sample containers and expired reagents. Extensive disposal operations have been conducted in the waste characterizations and extractions laboratory. As a result, these laboratories hoods have been cleared of samples and waste containers.
- (6) The auditor noted that waste storage areas are cluttered and need general housekeeping. Since the audit, Paragon has disposed of the samples in the waste characterization laboratory. This disposal effort has provided more bench space in the laboratory areas and reduced clutter. In addition, Paragon has scheduled and completed three (3) pickups of radioactive wastes since the audit. Seven (7) barrels of low level radioactive waste water and one (1) barrel of mixed hazardous/low level radioactive waste water been removed from the 90 Day Accumulation Area. Finally, Paragon has assigned "management functions" to individuals and groups that address housekeeping duties throughout the facility.

Corrective Action	Date of Completion	Responsible Parties
Boxes and equipment removed from hallways. Laboratories cleaned and reorganized.	02-15-00	H&S Department
State of Colorado Department of Health and Environment verifies that multiple,	02-21-00	H&S Department

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small containers in SAA acceptable (55 gallon maximum, each kind of waste).		
Housekeeping responsibilities increased for staff and cleaning crew throughout facility.	02-22-00	H&S Department
Housekeeping responsibilities increased for staff and cleaning crew in Sample Receiving Area	02-22-00	H&S Department
Removal of waste containers, sample containers, and chemical reagents from hoods and bench tops	02-15-00	H&S Department
Waste disposal (3 pickups), housekeeping functions assigned	02-15-00	H&S Department

Item SH-991207-B: *A program for periodic chemical exposure monitoring has not been defined (Priority II) (29 CFR 1910.1450).*

Response SH-991207-B: Paragon is developing a comprehensive chemical exposure monitoring program in order to address this finding. The monitoring program will include a combination of passive air samplers that will be sent to a NIOSH-approved laboratory for analysis and direct reading measurements taken in the laboratories. An SOP for the Chemical Exposure Monitoring Program will be developed. Comprehensive monitoring throughout the laboratory will be performed yearly. Attached for your review please find the Sampling and Analysis Plan for Laboratory Chemical Exposure Monitoring (*Attachment SH-991207-B*).

Corrective Action	Date of Completion	Responsible Parties
Develop sampling plan for chemical exposure monitoring at PAI	02-24-00	H&S Department
Order passive and direct samplers	03-20-00	H&S Department
Develop sampling record forms	02-25-00	H&S Department
Develop chemical exposure monitoring database	02-25-00	H&S Department
Perform monitoring	04-15-00	H&S Department
Send passive samplers to be analyzed	04-15-00	H&S Department
Evaluate and record data	06-01-00	H&S Department
Prepare chemical exposure monitoring SOP	04-30-00	H&S Department

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Item WM-991207-A: *Potentially radioactive sample processing waste is not segregated from sanitary trash. (Priority II) (10 CFR 20, Oak Ridge Site Specific Requirements)*

Response WM-991207-A: In order to correct this situation, Paragon is developing and implementing a contact waste management system. To date, the two (2) waste management SOPs have been revised. SOP 003 entitled "Non Radioactive Waste Disposal" has been revised to include contact waste management for hazardous waste. The SOP 015 has also been revised to include contact waste management for radioactive waste. In addition, the laboratories have been provided with dedicated, labeled containers for collections of (1) RCRA hazardous contact waste, (2) low level radioactive contact waste, and (3) mixed hazardous radioactive contact waste. These containers are sealable, 5-gallon containers. After the containers have been filled, they are transported from the SAA to the 90-day storage area. Attached for your review please find SOP 003 and 015 and supporting documentation from in-house training sessions (*Attachment WM-991207-A*).

Corrective Action	Date of Completion	Responsible Parties
Provide a contact waste collection system for all laboratories	12-30-99	H&S Department
Revise SOP 003 to include contact waste management	02-03-00	H&S Department
Revise SOP 015 to include contact waste management	02-03-00	H&S Department
Develop training program for contact waste management	03-10-00	H&S Department
Provide training on contact waste management	03-23-00	H&S Department

Item WM-991207-B: *PCB wastes are not managed in compliance with the Toxic Substances Control Act (TSCA). (Priority II) (40 CFR Part 761).*

Response WM-991207-B: Paragon has taken several steps to address this finding. First, Paragon has revised and combined three (3) hazardous waste management SOPs into SOP 003, entitled "Non Radioactive Waste Disposal." The revised SOP addresses the management of PCB wastes as required by 40CFR Part 761.

- (1) The auditor noted that waste containers used to accumulate PCB wastes do not display the TSCA required labels. Paragon has labeled the PCB containers with the EPA-mandated label: "**Caution Contains Polychlorinated Biphenyls.**"

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- (2) The auditor noted that PCB wastes are accumulated ~~without ensuring~~ that the TSCA required storage time limits are met. The container in the GC-HPLC lab has been designated as a temporary 30-day storage container. The container will be transferred to the 90-day area on a monthly basis. Please note that this material also contains RCRA hazardous wastes; therefore, this material may only be stored on site for 90 days after it leaves the temporary storage area (in contrast to the one (1) year on-site storage allowed for PCB only waste).
- (3) The auditor noted that there is no one-year storage area for PCB wastes at Paragon. It is true that Paragon does not have a one (1) year storage area for PCB wastes, because we do not collect PCB *only* wastes, as discussed above.
- (4) The auditor noted that there is no documented method for sharing information regarding PCB results greater than 50 ppm to other laboratory areas. Paragon's LIMS system is being programmed to assist with several aspects of hazardous waste management. One feature is a bulletin board warning message that will appear on all LIMS terminals for any sample that contains PCBs that have an aggregate Aroclor (PCB) concentration greater than 50 ppm. This warning message will appear on all terminals as soon as the data are entered into the LIMS, thereby enabling proper disposal in a timely manner.
- (5) The auditor noted that Paragon has not conducted audits of our two major waste TSD vendors, Permafix and Clean Harbors. In order to address this finding, Paragon conducted an audit of Clean Harbors Kimball, Nebraska Temporary Storage & Disposal Facility (TSDF) on 02-17-00. Paragon will conduct an audit of the Perma-Fix Environmental Services facility in Gainesville, Florida by 07-15-00. Attached for your review please find a copy of Paragon's audit of Clean Harbors (*Attachment WM-991207-B*).

Corrective Action	Date of Completion	Responsible Parties
Correct labeling deficiencies on PCB collection containers	01-10-00	H&S Department
Incorporate PCB waste management requirements into SOP 003	02-03-00	H&S Department
Set up 30 day transfer schedule for PCB temporary storage area	02-10-00	H&S Department
Implement long term storage area (90 Days) for PCB wastes	02-10-00	H&S Department
Enhance LIMS to include bulletin board notification of PCB concentrations greater than 50 ppm	05-15-00	H&S Department
Conduct audit of Clean Harbors in Kimball, NE TSDF	02-17-00	H&S Department

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Conduct audit of Perma-Fix Environmental Services in Gainesville, FL TSDF	07-15-00	H&S Department
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Item WM-991207-C: *Waste containers used for storage of analytical wastes containing triethylamine are of inadequate integrity. (Priority II) (40 CFR Parts 262.34; 265.171-172).*

Response WM-991207-C: Paragon has addressed this finding by transferring all triethylamine wastes to 15 gallon UN1A1 steel drums, labeled with a uniform hazardous waste label, annotated as "waste triethylamine." A waste profile for this material was developed with Clean Harbors for incineration at Kimball, Nebraska. Attached for your review please find a copy of the waste profile (**Attachment WM-991207-C**).

Corrective Action	Date of Completion	Responsible Parties
Transfer triethylamine wastes to UN Specification 1A1 Drum	02-05-00	H&S Department
transport triethylamine wastes to Clean Harbors for incineration	04-05-00	H&S Department

Item WM-991207-D: *The PAI waste management plan does not reflect current practices and is not adequate in describing many ongoing waste processing activities. (Priority II) (DOE Order 414.1, ICPT Terms and Conditions).*

Response WM-991207-D: The SOPs that address waste management have been revised. SOP 003, entitled "Non Radioactive Waste Management," has been rewritten and information from SOPs 004 and 006 has been incorporated. SOPs 004 and 006 have been retired. The revised SOP 003 addresses: waste characterization, container management, waste types managed, satellite accumulation area (SAA) management, 90 day accumulation area management, mixed waste management, and PCB management. The SOP 015 entitled "Disposal of Radioactive Waste" has also been rewritten. Attached for your review please find a copy of SOPs 003 and 015 (**Attachment WM-991207-A**).

Corrective Action	Date of Completion	Responsible Parties
Revise and merge hazardous waste SOPs 003, 004, 006	02-03-00	H&S and QA Departments
Rewrite radioactive waste disposal SOP 015	02-03-00	H&S and QA Departments



Item WM-991207-E: *The process for disposition of samples that have exceeded their archival date is not adequately documented or implemented. (Priority II) (ICPI Terms and Conditions).*

Response WM-991207-E: Paragon acknowledges that a backlog of old samples were awaiting disposal at the time of the audit. PAI has dedicated two full-time employee equivalents to perform sample disposal for the past six (6) months. Since your visit, approximately 90% of the samples in the upstairs storage area have been disposed.

In addition, we have built a 16' by 25' archived sample storage room in the radiochemistry laboratory common area. This area provides an organized, consolidated storage area for archived samples, which eliminates the need for additional archived sample storage areas throughout the facility. The area accommodates five 8'x10'x4' shelving units. The samples coming out of in process storage are organized according to archive expiration date and waste disposal type.

Paragon's IS staff is programming the LIMS to enable us to manage waste disposal records in an automated fashion. This LIMS waste module will track disposal of samples electronically within the LIMS and will assist in the classification of samples into the appropriate waste stream. The manual system for documenting disposal records will be used until the LIMS module has been completed. The backlog of sample disposal records has been compiled and organized by work order.

Corrective Action	Date of Completion	Responsible Parties
Sample disposal of entire archived sample backlog	05-01-00	H&S Department
Construction of archived sample storage room	02-01-00	H&S Department
Implementation of LIMS hazardous and radioactive waste management module	06-01-00	IS Department
Delegation of current paper sample disposal record processing to Reports Management staff	02-25-00	H&S Department
Compilation of paper sample disposal records	04-01-00	Reports Management Department

Item WM-991207-F: *PAI is not performing bi-annual (sic) reviews of waste profiles as required. (Priority II) (PAI SOP.)*

Response WM-991207-F: Paragon agrees to internally re-characterize our waste streams on a biennial schedule. Samples will be taken randomly and analyses performed as described in The Sampling and Analysis Plan for Hazardous Waste Streams. The

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waste profiles will be modified as appropriate based upon the analytical results. For your review please find the Sampling and Analysis Plan that describes Paragon's approach to re-characterizing our waste streams. (*Attachment WM-991207-F*).

Corrective Action	Date of Completion	Responsible Parties
Develop Sampling and Analysis Plan	02-25-00	H&S Department
Sample waste streams: aqueous lab waste; acidic aqueous lab waste; contaminated soils and solids; halogenated waste; non-halogenated waste; discarded extract vials; PCB/RCRA debris; and oil and solvent waste	04-10-00	H&S Department
Submit samples to laboratories for analyses	04-10-00	H&S Department
Analyze and report samples	05-15-00	operations
evaluate analytical data	06-15-00	H&S Department
Revise waste profiles as necessary	07-01-00	H&S Department

Item RC-991207-A: *The process for identifying incoming samples that require a prescreen for radioactivity analysis is informal. (Priority II) (ICPT Terms and Conditions).*

Response RC-991207-A: Paragon believes that our process for identifying incoming samples that require a prescreen for radioactivity is well defined and thoroughly documented. Paragon's Project Managers work with clients to define all technical and service requirements prior to receipt of samples. This interview includes questions about potential radioactivity (e.g., site history, historical data, expected radionuclides and levels of activity). Project Managers distill project requirements to all Sample Receipt and Operations personnel by issuing an Incoming Project Notice (IPN). This notice is generated through the LIMS and addresses health and safety and waste disposal information -- including prescreen requirements. The Sample Receiving staff determines which sites/samples require prescreen from this information. In the event that samples arrive unannounced, the Sample Receiving staff place the samples on "hold" status and forward Chain of Custody information to the Operations Manager. The Radiation Safety Officer and Operations Manager assess the new client's prescreen requirements via a teleconference with the client.

In general, Paragon performs a prescreen for radioactivity on all samples received from DOE sites or on behalf of a DOE site, unless the client provides reliable prescreen data.

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At the time of the audit, the auditor noted a list of clients and sites that require prescreening for radioactivity. In order to update our list to include all ICPT sites/projects, Paragon respectfully requests that DOE ORO provide a list of all potential sites, projects, and contractors. In the event that an ICPT contractor sends samples to Paragon without a signed contract in place, our routine administrative controls will address the prescreen concern.

Corrective Action	Completion Date	Responsible Party
Provide comprehensive list of ICPT DOE sites, projects, and contractors from DOE ORO	04-30-00	DOE ORO
Update internal list of clients and sites that require prescreen analysis	05-15-00	H&S Department

Item RC-991207-B: *Radioactive sample shipments are not surveyed for internal surface contamination before sample handling. (Priority II) (10 CFR 20).*

Response RC-991207-B: PAI will institute a removable contamination survey program for sample containers that contain radioactive material shipments. The types of shipments that will undergo sample container removable radioactive material contamination surveys include: excepted radioactive material packages, low specific activity packages, and radioactive I, II or III packages. The most common types of packages to be received at Paragon are excepted radioactive material and radioactive I packages. The sample containers will be subjected to a composite removable radioactive material contamination survey (swipe). The swipe will be counted for 5 minutes by both the Ludlum 1000 Scaler with 43-10 Alpha Scintillation Detector for detection of alpha particles and the Ludlum 1000 Scaler with 44-7 Geiger-Mueller Detector for detection of Beta/Gamma emissions. Action levels are 20 dpm/100 cm² removable alpha and 200 dpm/100 cm² removable beta/gamma (Nuclear Regulatory Commission's Decommissioning Release Limits for Unrestricted Use). If the composite sample composite removable radioactive material contamination swipe results are less than the above limits, the samples will be released. If the limits are exceeded on the composite swipe, then all containers must be swiped. The sample receiving staff will be trained to perform composite removable radioactive material swipes on sample containers and to evaluate results.

Corrective Actions	Date of Completion	Responsible Parties
Prepare incoming sample removable radioactive material contamination survey log form	03-25-00	H&S Department

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Train sample receiving staff to prepare incoming sample removable radioactive material contamination survey	04-15-00	H&S Department
Revise SOP 208 to include incoming sample removable radioactive material contamination survey	04-05-00	H&S Department

Item RC-991207-C: Radiological area surveys do not include periodic monitoring for fixed contamination or airborne contamination. (Priority II)(10CFR20)

Response RC-991207-C: Paragon will perform monthly monitoring throughout the laboratory for fixed radioactive material contamination. The sampling areas may be viewed in the Monthly Fixed Radioactive Material Contamination Survey Form (*Attachment RC-991207-C*). The surveys will be conducted using an NE Electra with a DP6B Dual Alpha /Beta Scintillation Probe. The action levels for the Fixed Contamination Monitoring will be 25% of the United States Nuclear Regulatory Commission's Unrestricted Release Limits for Fixed Radioactive Material Contamination. 50 dpm/100 cm² alpha and 250 dpm/100 cm² beta/gamma). The first set of fixed contamination measurements will be completed by 02-29-00.

PAI will monitor the laboratories for airborne particulate radionuclide contamination on a quarterly frequency. The air samples will be taken using an SAIC Radeco Particulate Air Sampling Pump. The sampling volume will be large enough to give an MDA that is at least 5% of the 10CFR Part 20 Derived Air Concentration (DAC) Limit.

Corrective Actions	Date of Completion	Responsible Parties
Prepare Survey Locations and Fixed Radioactive Material Contamination Survey Forms For Monthly Fixed Radioactive Material Contamination Monitoring	2-21-00	H&S Department
Implement Monthly Fixed Radioactive Material Contamination Survey	2-29-00	H&S Department
Develop Sampling Plan For Airborne Monitoring For Radionuclide	2-29-00	H&S Department
Complete Air Sampling	4-01-00	H&S Department
Review and Evaluate Analytical Data	5-01-00	H&S Department

Observations

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Item 1: *The pipettes used to perform pH checks of incoming samples are stored in an open top box. Since these pipettes are immersed directly in the incoming samples to obtain an aliquot for pH checking, they should be covered to prevent dust and other contaminants from affecting these pipettes.*

Response 1: Paragon conducted a labwide training session on 02/17/00. Item #3 of this training addressed proper storage conditions for pre-cleaned materials that would preserve their cleanliness and prevent sample contamination. Direction was given to store the pipettes in their original box with the lid intact and to close the box when not in use. Attached for your review please find a copy of this training documentation (*Attachment Observation 1*).

Item 2: *The logbook used in sample receiving to record source check information for the ion chamber survey meter does not include a reference to the unique identification number of the source check standard.*

Response 2: The source is a qualitative check source and a unique identification number was not assigned by the manufacturer. On 02-16-00, Paragon assigned a unique identification number to the source (CSC1). Attached for your review please find a revised page from the Ludlum Model 3 Logbook that includes the unique identification number for the source check standard (*Attachment Observation 2*).

Item 3: *The list of emergency contacts is not posted by the telephone in the Hazardous Waste Storage Area.*

Response 3: Paragon has addressed this finding by posting names and telephone numbers of emergency contacts beside the telephone on 12-24-00.

Item 4: *A check source is not available for the rad survey meter used in the volatile organic analysis storage laboratory. The performance of rad survey instrumentation should be monitored with a source prior to use. The source check and the background readings should be documented.*

Response 4: Paragon will obtain check source for the survey meter and will create a logbook to document background reading. The H&S Department is responsible for acquiring the check source and creating the logbook by 04-30-00.

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Item 5: SOP 512, Revision 4, "Refrigerator Storage Blanks" should be placed in the SOP manual of the Volatile Organics laboratory. A copy of the SOP was located in the satellite SOP manual.

Response 5: Paragon concurs that each laboratory should have a controlled copy of every SOP needed to perform their job duties. The QA Department provided a controlled copy of SOP 512 to the Volatile Organics Laboratory.

Item 6: The current LQAP should be updated to contain the method requirements for SW-846 Method 8260 for the analysis of volatile organics by GC/MS. The LQAP contains the requirements for SW-846 Method 8240 which has been discontinued by the EPA.

Response 6: Paragon is aware that Method 8240 has been discontinued by the EPA. Paragon will delete references to this method in the next revision of the LQAP.

Item 7: The analysis of the refrigerator storage blanks must be analyzed within the 12-hour period following the injection of BFB. Paragon has an agreement with one of its clients to analyze the refrigerator storage blanks outside of the 12 period (sic). For all work associated with DOE-ORO, the analysis of all refrigerator storage blanks must be performed within the 12 period (sic).

Response 7: Paragon has changed its practices and revised SOP 512 to comply with this requirement. SOP 512, Revision 5 follows for your review (*Attachment SC-991207-A*).

Item 8: Refrigerators and freezers should be labeled to state that no food should be stored with samples.

Response: 8: All refrigerators, coolers, and main entrances into the laboratories have been labeled with signs as follows: "NO FOOD OR DRINK ALLOWED IN THIS AREA". This task was completed on 12-24-99.

Item 9: For DOE-ORO analyses of PCB analytes, Paragon should calibrate for Aroclor 1268. At the present time, Paragon does not include Aroclor 1268 in the analyte list.

Response 9: As of this writing, none of Paragon's clients has requested Aroclor 1268; therefore, Paragon has not included this Aroclor in its calibration scheme for Method 8082. Paragon will calibrate for Aroclor 1268 for DOE-ORO samples.

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Item 10: *The glassware cleaning SOP or an operator aide should be posted in the organic glassware cleaning area.*

Response 10: Paragon revised and reissued the glassware cleaning SOP 334 on 02-17-00. Paragon posted glassware cleaning instructions at the glassware cleaning areas on 02-17-00. Attached or your review please find SOP 334, Revision 2 (**Attachment Observation 10**).

Item 11: *The COC logbook for the GC laboratory has not been review (sic) since 1998. The logbook should reviewed (sic) by a supervisor on a routine basis.*

Response 11: Paragon concurs that this, and all, logbooks should be reviewed on a monthly basis. This practice has been addressed throughout the laboratory, as discussed in Item QA-991207-B. Attached for your review please find pages from this logbook that demonstrate a recent review (**Attachment Observation 11**).

Item 12: *The documentation of the daily maintenance for the GC instrumentation is being recorded in the daily runlog. A separate logbook should be used for the preventive maintenance documentation.*

Response 12: Per the auditor's request, Paragon has created a separate logbook that is dedicated to the documentation of preventive maintenance. Attached for your review please find a copy of a page from the new logbook (**Attachment Observation 12**).

Item 13: *The ICV daily working standard used for ion chromatography is prepared by diluting a secondary source; however, this dilution is not documented.*

Response 13: Paragon has edited the standards preparation information to include dilution information. Attached for your review please find a page from the revised standards preparation logbook (**Attachment Observation 13**).

Item 14: *Inorganic logbooks containing taped entries do not have a verification signature to reveal where the entry begins.*

Response 14: Paragon addressed the requirement for signing and dating logbook pages in the laboratory-wide training session on 03-13-00. Documentation of training and relevant SOPs are included in **Attachment QA-991207-B**.

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Item 15: *TOC and TOX standard are being stored in the metals sample refrigerator RU18. A separate refrigerator should be maintained for standard and sample storage.*

Response 15: In December 1999, Paragon purchased a separate refrigerator for the storage of TOC and TOX standards. Attached for your review please find a copy of a logbook page from the new standards refrigerator (*Attachment Observation 15*).

Item 16: *Oak Ridge Sample Management Program requirements for Tritium analysis specify that a refrigerator blank must be stored, distilled and counted along with the samples.*

Response 16: Following receipt of the Oak Ridge QAPjP / Terms and Conditions, Paragon's Project Manager will discuss this requirement during program specification.

Thank you again for your time and assistance during the on-site audit. We hope that our responses meet your requirements. Please contact me at 970 490 1511 if additional information is required and I will be glad to provide it.

Respectfully Submitted,

Debra Henderer
Quality Assurance Manager
Paragon Analytics, Inc.

Enclosures



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December 18, 1999

Mr. David Bourne
Department of Energy
Albuquerque Operations Office
Environmental Restoration Division
P.O. Box 5400
Albuquerque, NM 87115

**RE: DOE-AL Characterization Management Program Audit of Paragon Analytics, Inc.
October 12-15, 1999**

Dear Mr. Bourne:

I am writing to respond to Mr. Minter's audit report of October 25, 1999. Paragon sincerely appreciates the DOE-AL on-site audit of our systems and processes and the time spent with our employees. We are pleased to respond to the 13 observations from the audit. Paragon's responses and corrective actions follow for your review. At Mr. Minter's direction, Paragon has forwarded enclosures to his attention *only*.

Observation 1:

Paragon QA staff members are currently revising the Quality Assurance Plan. The following items were not addressed or not adequately addressed in the new plan.

Item 1) The QAP section on analytical procedures does not address SOP content or format.

Response 1) Paragon has revised Section 6 of the Laboratory Quality Assurance Plan (LQAP) to address SOP content and format. Paragon has revised the form that addresses content and format of SOPs (Form 154). In summary, the LQAP and Form 154 prescribe: a three-level review for all SOPs prior to release; 12 sections (e.g., Scope and Application, Procedure, Quality Control, References); a summary table of internal quality control procedures and corrective actions (e.g., quality control check, frequency, acceptance criteria, corrective action). Please see *Attachment 1* for the revised LQAP pages from Section 6 and Form 154.



Item 2) The training requirements in the QAP and training SOP do not specify what general training is required prior to starting different types of work. For example, those documents do not require that QA indoctrination and radiation safety be completed before starting work.

Response 2) Paragon has revised Section 14 of the LQAP and SOP 329 to address required training prior to beginning work at Paragon. Paragon requires that each employee receives a four part orientation before working in the laboratory: human resources; quality assurance; health and safety and radiation safety; and department orientation. Paragon further requires that each employee successfully complete health and safety and radiation safety tests and an IPR study before working in the laboratory. Please see *Attachment 1* for the revised LQAP pages from Section 14 and SOP 329, Revision 2.

Item 3) The section that discusses MDL studies does not specify the minimum number of measurements required.

Response 3) Paragon has revised Section 3 of the LQAP and SOP 329 to specify the minimum number of measurements required for an MDL study. Paragon requires that a minimum of eight (8) replicates be performed for each MDL study. Please see *Attachment 1* for pages from Section 3 of the LQAP and SOP 329, Revision 2, Section 5.

Item 4) The sections and tables for instrument calibrations and standards do not provide comprehensive guidance. For complete standards protocols, see section 2.7.4 of the Model SOW.

Response 4) Paragon has revised SOPs 300 and 734 to ensure compliance with section 2.7.4 of the Model SOW. Please see *Attachment 1* for SOP 300, Revision 5 and SOP 734, Revision 5.

Observation 2:

The MDL studies for several methods were reviewed. Paragon does perform MDL studies on the confirmation columns for GC methods that require confirmation. However, the laboratory conducts the second-column MDL studies for 8330 with mixtures of compounds, and when analyte peaks cannot be resolved no detection limits are calculated.

Response 2) Paragon performs annual MDL studies for every matrix, method, instrument, and analytical column as required by SW-846 and 40CFR Part 136 Appendix B. In reporting an MDL value for a given method and matrix, Paragon chooses the highest calculated MDL value (if multiple values are available from different instruments and/or columns).

For gas and liquid chromatography methods, coelution may occur for single component compounds. Historically, Paragon has accepted this coelution as a limitation of the methodology -- provided that the higher calculated MDL value is less than the required reporting limit. As requested, Paragon agrees to perform additional MDL studies using separate spiking mixtures or to provide standard verification data in order to verify that coeluting compounds can be chromatographed on both analytical columns.



Observation 3:

A general review of laboratory SOPs was done. Several SOPs did not reference the associated regulatory methods, and some did not fully meet the specifications of the methods that were referenced. An example is that the TOC SOP does not require quadruplicate analyses for method 9060. Note: Paragon staff members are currently in the process of reviewing and revising many SOPs.

Response 3) Paragon has revised the form that addresses content and format of SOPs (Form 154) to ensure consistency in format and content when writing and/or revising analytical SOPs (e.g., referencing regulatory methods in the title, Section 1 / Scope and Application, and Section 12 / References). Please see **Attachment 1** for Form 154. Further, Section 10 of Paragon's analytical SOPs is entitled "Deviations From Method" and addresses Paragon's requirement to disclose and discuss method discrepancies/deviations.

SOP 803, "Analysis of Total Organic Carbon by Methods 415.1 and SW9060" is currently being revised. Revision 4 will address the quadruplicate analyses required by SW Method 9060. Debra Henderer and Darryl Patrick are responsible for completing Revision 4 by December 31, 1999. A copy of the revised SOP is available upon request.

Observation 4:

Paragon's GALP practices are generally consistent with Model SOW requirements. However, there is little documentation on the computer operations. Also, QA generally does not include the computer systems in the internal audit program.

Response 4) Paragon acknowledges that documentation of computer operations can be augmented. Confidentiality and distribution of proprietary information are concerns; therefore, Paragon does not provide all available documentation of computer operations to clients or auditors. Several relevant SOPs currently exist in *draft* form (e.g., LIMS Version Control, Software Validation, Backup and Restoration Protocols -- which includes archiving of Backups). Copies of the *draft* LIMS Version Control SOP, Software Validation, and *draft* Backup and Restoration Protocols SOP are enclosed as **Attachment 4**. All computer operations SOPs are scheduled to be completed and released by February 25, 2000. Glenn Barrows, Manager of the IS Department, is responsible for coordinating this effort.

Storage and retrieval of outdated software versions are addressed in the attached Computer Operations Policy Statement as **Attachment 4**. Paragon Form 52, mentioned in the Statement, is also provided for your review. Please note that LIMS iterations, which are developed in-house, *are* formally tracked and archived as discussed in the attached *draft* LIMS Version Control SOP. The LIMS database tracking practice addresses the audit concern of "recording of implementation dates for new software."

Storage of instrument operation parameter files is addressed by the attached *draft* Backup and Restoration Protocols SOP. Instrument parameter files are saved to the hard drive of the



computer controlling the instrument and are, therefore, a component of the periodic instrument PC backup.

Retention of software manuals and user instructions are also addressed in the attached Computer Operations Policy Statement. Manuals for LIMS applications are proprietary information and are currently being revised to incorporate recent upgrades.

Per the auditor's request, Paragon has included several computer-related operations to the schedule of internal audits performed by the QA Department. Please see *Attachment 4* for the Schedule of Internal Audits.

Observation 5:

The laboratory defines LCS as a spike of a clean matrix. However, laboratory control samples are intended to test the efficacy of the entire analytical process, including the digestion steps. Spikes are soluble by definition, deriving from standard solutions, and can generally be recovered with no digestion at all. In practice, Paragon does use solid reference materials in many analyses. Paragon staff members point out that some clients specify recovery limits that are precluded by the inadequate homogeneity of the SRMs.

Response 5) Paragon has rewritten Section 9 of the LQAP to clarify the definition of an LCS and address the auditor's requirement to use solid reference materials (SRMs) if available for metals and radiochemistry. Paragon notes that we require SRMs to be purchased from an NIST-approved vendor, if available. In addition, Paragon accepts the vendor's control limits. A copy of the revised LQAP pages from Section 9, sample metals SRM limits, and Paragon's agreement with LANL are included for your review as *Attachment 5*.

Observation 6:

A cursory review of QC control limits was done. The acceptance limits for the surrogate recovery of 2,4,6-tribromophenol were given as 0 to 123%; however, the control chart data indicated that acceptance limits of 42 to 123% were more appropriate. This specific example suggests that a general review and update of Paragon's QC control limits is warranted.

Response 6) Paragon is updating intralaboratory qc limits for all methods and matrices and for all target compounds and surrogate compounds. We are evaluating all data points for LCS/LCSD samples entered into our LIMS system since January 1999. Following review and approval of data, the QA Manager will distribute revised qc limits to operations and intralaboratory qc limits will be amended in the database to ensure that data are evaluated against the appropriate limits. We anticipate that review and distribution will be completed by January 15, 1999. Please see *Attachment 6* for samples of initial qc data and the memorandum that will be distributed to all employees.



Observation 7:

The issues discussed below were identified during a brief review of several data packages.

Item 1) The SOP field on several of the worksheets was left blank or was filled in with NA. All analytical work must reference the SOP and revision numbers that are applicable to that work.

Response 1) Debra Scheib of the QA Department conducted a training session for technicians and analysts on November 16, 1999. This training focused on completion of benchsheets by technicians and analysts and supervisory review of benchsheets. A copy of the training sign-off sheet follows for your review (**Attachment 7**).

Item 2) The GC and HPLC run logs were missing several of the required entries listed in section 3.2.1 of the Model SOW. Our review of worksheets and run logs for other areas showed that Paragon's analysis documentation is generally very good. However, we noted several cases for which the instrument used, SOP, and/or calculations were not given. Paragon should conduct a general review of worksheets and run logs to ensure compliance with section 3.2.1 in all areas.

Response 2) Paragon has conducted a review of GC and HPLC forms (worksheets, run logs). Software constraints are being investigated to determine if the following information may be incorporated into the header of the instrument's computer-generated runlog: method reference, SOP/Revision numbers, and instrument ID. Peter Gintautas is responsible for implementing the (potential) software changes by December 31, 1999.

In addition, Form 410 has been developed to provide supplementary information. As indicated, this form will provide documentation of internal standards check, surrogate check, reanalysis requirement, and general comments. Form 410 was implemented on November 18, 1999. Please see **Attachment 7** for a copy of Form 410 and the associated training sign-off sheet. If the software cannot provide the header information, then Form 410 will be revised to include this information.

With regard to calculations, Paragon understands that the auditor's comment refers to the documentation of the computing of analytical results for all parameters, so that a data validator may recreate the results. By December 31, 1999, the QA Department will conduct training for each analytical group to ensure that the following information is recorded on runlogs: name of analyst who performed analysis; instrument name and unique ID used for analyzing samples; initials of reviewer; calibration information (e.g., date, data file names, statement of successful calibration); standards information (e.g., name, preparation date, expiration date); method reference; date and time of analysis. Paragon notes that the equations used to calculate reported values are provided in the individual case narratives and that sample-specific analytical factors appear on the Form Is generated by the LIMS system (e.g., initial volume, final volume, dilution factor).



Item 3) One of the packages included analytical data from a subcontract laboratory. However, this situation was not discussed in the narrative. Case narratives should discuss any subcontracted work, give the name and contact numbers for the contractor, and include a definitive statement that the subcontracted work was done with prior client approval.

Response 3) Paragon concurs that subcontracted analyses should be discussed in the cover letter. Paragon has created a template for LANL SMO cover letters that addresses: method subcontracted; name of subcontractor (representative and phone number available on data report); and statement regarding prior client approval. Please see **Attachment 7** for documentation of the template and supporting internal emails.

Item 4) The calibration and ICV standards used in GC GRO analyses were expired under both DOE-AL and Paragon protocols. Note: the work examined in this case was not LANL work, and we did not seek out documentation for GRO work performed for LANL.

Response 4) Paragon acknowledges that the standards observed were expired, partially as a result of misinterpretation of SOP 300, "Standards Preparation and Shelf Life." Paragon has revised SOP 300 to clarify requirements of the Model SOW. Controlled copies of Revision 5 have been distributed. Please see **Attachment 1** for a copy of SOP 300, Revision 5. Printouts for current GC/HPLC standards are provided for the auditor's review. The expiration dates shown for intermediate dilution GRO working standards now correctly reflect a one-month (30 day) duration. Please note that these standards have been replaced twice (i.e., immediately and one month later) since the October 12 audit.

Item 5) Randomly selected (not LANL) documentation for GC and HPLC work showed that standards used for calibration and ICVs were not specifically called out on the run logs. In addition, standard preparation information for the working solutions was not available in the HPLC work, some data were obliterated in error correction, and leading zeroes were not always used with numbers less than one.

Response 5) Nomenclature for calibration standards, both first and second source, has been reviewed and will be revised in conjunction with the implementation of the computerized standards management program discussed above.

Peter Gintautas conducted a training session regarding data correction practices and the use of leading zeroes on November 15, 1999. A copy of the training sign-off sheet is included for your review with **Attachment 7**.

Item 6) The run logs used in GC/MS work at Paragon are among the best we have seen. However, the run logs used in GC and HPLC work contain no comments fields and generally do not meet the criteria given on pages 22 and 23 (items 7 and 10) of the audit worksheet.

Response 6) As discussed above, Form 410 has been implemented to supplement the information shown in the run log (i.e., run sequence printout). In addition, software capabilities are being explored to determine if information such a method reference, SOP/Revision number,

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and instrument ID can be incorporated into the printout's header. Peter Gintautas will complete the investigation by December 31, 1999. If the additional information cannot be incorporated into the run sequence header, then Form 410 will be revised to include this information.

Observation 8:

The laboratory generally allows reanalysis of a CCV that fails. Although SW846 methods for organics allows this, SW846 methods 7000a(8.3), 6010b(7.4), and 6020(7.8 and 8.8.3) require reanalysis of all samples associated with a CCV failure.

Response 8: In practice, Paragon understands and complies with the CCV criteria stated in Methods 7000A, 7470A/7471A, and 6010B. For 7000 series and Method 6010B analyses, Paragon calibrates the instrument daily and analyzes a mid-range CCV after every 10 samples. Paragon requires that the CCV's value is $\pm 20\%$ of the true value for 7470A/7471A methods and $\pm 10\%$ of the true value for Method 6010B (SW-846 references: Method 7000A, Section 8; Methods 7470A/7471A, Section 8; Method 6010B, Section 8). For both AA and ICP methods, samples not bracketed by a compliant CCV are reanalyzed. Paragon understands that CCV failure requires recalibration of the instrument for metals analyses.

Paragon has revised SOPs 805 and 807, "Determination of Metals by Inductively Coupled Plasma Emission Spectroscopy -- Methods 6010B, CLP ILMO4.0, or 200.7" to clarify qc requirements related to CCV evaluation for radial and axial instruments, respectively. Please see **Attachment 8** for revised pages from SOP 805, Revision 2 (radial ICP) and SOP 807 Revision 5 (axial ICP).

Paragon has revised SOP 812, "Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy -- Methods 7470A..." to clarify qc requirements related to CCV evaluation for AA instrumentation. Please see **Attachment 8** for revised pages from SOP 812, Revision 7.

Paragon does not perform Method 6020; therefore, no SOP revisions are warranted.

Paragon understands that the auditor's observation regarding evaluation of a CCV applies to all inorganic methods. Darryl Patrick is responsible for reviewing all inorganics SOPs for method compliance with respect to CCV evaluation by December 31, 1999.

Observation 9:

A general review of the laboratory training records revealed that most of the files are missing one or more records. Also, the required training is not formally defined for the analysts (see Observation 1). Note: The laboratory has recently implemented tracking systems for training and has developed a plan to review and update training records.

Response 9) Paragon acknowledges that training records are not yet complete for every employee. As stated, the QA Department has developed a matrix that lists required training/documentation and tracks the documentation of each employee's training. The QA

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Department has also developed an initial "QA orientation" package that each employee must complete before performing any analyses. The QA Department will continue to update every employee's analytical, quality, and LIMS training files and The Health and Safety Department will continue to update every employee's health and safety and radiation safety training files. Current employees' files will be updated by March 01, 2000. Examples of method/SOP, QA, LIMS, health and safety, and radiation safety training documentation follow for your review as *Attachment 9*.

Observation 10:

The issues listed below apply to radiochemistry.

Item 1) Paragon uses small paint cans, a paint shaker, and steel balls to pulverize about 30 to 50 grams of sample for radionuclide analyses. LANL has approved this practice in the past. However, Paragon's practices deviate from the soil preparation requirements given in the Model SOW, and hence LANL's next SOW. The laboratory should be aware that either process changes or acquiring new formal permission to deviate will be required if Paragon participates in the next LANL contract. No response is required from Paragon for this item.

Item 2) Paragon does not have a procedure for performing salt fusions in radionuclide sample preparation. While not required in general for DOE-AL facility work, the laboratory should be aware that GJPO does require salt fusions. Procedures to address this issue must be developed if Paragon performs radionuclide determinations for GJPO. No response is required from Paragon for this item.

Item 3) The calculations for analytical results, detection limits, and uncertainties are not included with radiochemistry data at Paragon. Since these calculations are lengthy, we accept calculation summary inserts in data packages in lieu of presenting them with the documentation for each analysis.

*Response 3) Paragon has summarized calculations for the most frequently requested radiochemical analyses (e.g., gross alpha/beta, ²²⁶Ra, ²²⁸Ra, ⁹⁰Sr, ³H, alpha isotopics). Paragon agrees to provide these summaries with Level IV data packages for DOE Albuquerque facilities. Copies of the calculations are provided as *Attachment 10*.*

Item 4) The GFPC calibration practices at Paragon showed several deficiencies. Paragon chemists generate mass attenuation curves on only one of the GFPC detectors. Their practice is to then run 3 of the calibration standards on each of the other detectors to verify that the calibration data will work for those detectors.

Item 4a) Curves should be generated for all detectors. If this is not done, all of the calibration standards should be run on every detector and some averaged curve developed. However, if Paragon chooses this latter approach, reasonable and specific acceptance criteria must be developed and adhered to.

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Response 4a) Paragon is revising our calibration procedures to incorporate the auditor's observations. Recalibration of the LB4100 is scheduled for the first week of December 1999. The full set of absorption standards will be acquired for each detector and a curve evaluated for each detector. If the data are reproducible, data may be averaged to yield a single curve for all detectors. Single fitted points may not deviate more than 10% RPD from fitted points and the goal for average deviation will be 10%. Each curve consists of several points (8-16 points) of relatively high level standards. As a result of geometry considerations and uneven deposition of precipitate on planchets, a single standard may not fit the curve. In this case, Paragon will discard a particular standard that does not fit the curve and re-evaluate the curve. Dave Burns and Bob Shannon are responsible for completing the recalibration of the LB4100 by December 15, 1999.

Item 4b) The practice of running 3 calibration standards on the uncalibrated detectors is not discussed in Paragon's LQAP or procedures. All such calibration activities should be formalized in laboratory documents.

Response 4b) Paragon has revised Sections 7 and 13 of the LQAP to address calibration activities as discussed in Response 4a above. Please see *Attachment 10* for relevant pages from Sections 7 and 13 of the LQAP.

Item 4c) For the most recent work, only two calibration standards were run on the uncalibrated detectors instead of three. Clearly, the current practices were not adequately conveyed to staff. This suggests a need for better training and review processes.

Response 4c) Paragon concurs that additional training is required for GFPC calibration practices. Following recalibration of the LB4100, Bob Shannon will conduct a training session for analysts. This training will be documented and completed by December 31, 1999.

Item 4d) For the most recent work, the calibration standards read back with errors of from 8 to 15 percent on several of the uncalibrated detectors. These were high-activity standards, and hence these unacceptably large errors were in fact individual efficiency as opposed to counting error.

Response 4d) Paragon acknowledges these concerns. We believe that recalibration of the LB4100 and training of analysts (as proposed above) will address concerns raised in Items 4b-d.

Item 5) Many of the radiochemistry analyses do not include second-source standards. Paragon should acquire and use second-source standards for the analyses that now have none (see Model SOW section 3.6.9).

Response 5) Paragon concurs that second-source, NIST-traceable standards should be used throughout the laboratory to verify calibration of instruments by primary NIST-traceable standards. Paragon notes that reverification of radiochemistry standards is an acceptable practice. Paragon agrees to address this oversight that was detected in the Radiochemistry Department. Dave Burns and Bob Shannon are responsible for ensuring that second-source standards are ordered and verified by December 31, 1999, where NIST-approved sources are

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available and cost is not prohibitive. Please see the memorandum that addresses this requirement (*Attachment 10*).

Paragon notes that we participate in the following performance evaluation radiochemistry studies: US EPA NERL (now replaced by the NIST-approved vendor, ERA); US DOE MAPEP; and US DOE EML. Paragon's continued excellent performance in all three (3) studies demonstrates our accurate calibration and resultant quantitation. Results of all performance evaluation studies follow as *Attachment 10*.

Item 6) While Paragon's counting instrument calibration practices are compliant except as noted above, the related specifications in LQAP are often incomplete or incorrect. Paragon should carefully revise the LQAP to correctly and comprehensively describe the calibration actions and the frequency with which they will be performed.

Response 6) Paragon has revised Sections 7 and 13 of the LQAP to fully describe the calibration requirements for radiochemistry analyses. Please see *Attachment 10* for Sections 7 and 13 from the LQAP.

Item 7) Paragon's tracer and carrier recovery acceptance criteria are compliant with the current LANL SOW, but are not compliant with the Model and future LANL SOWs. The laboratory should be aware that a change will be necessary to achieve compliance if Paragon participates in the next LANL contract. No response is required from Paragon for this item.

Item 8) As noted in the Overview section, all of the applicable radiochemistry analyses at Paragon correctly include sample-specific correction for chemical recovery. However, Paragon performs a chemical separation and analyzes ^{226}Ra by gamma spectroscopy using a gravimetric barium measure of recovery. Unfortunately, Paragon does not do Lucas cell alpha scintillation determinations at present. When using gravimetric barium as a recovery measure, indigenous barium can interfere and cause results to be biased low. We request that the laboratory consider using a ^{133}Ba tracer in this analysis.

Response 8) Paragon's current practice of measuring the pre- and post-separation concentration of barium by ICP-AES provides accurate quantitation of ^{226}Ra and overcomes the possible low bias that native barium may cause. Paragon understands that a matrix spike must be prepared and analyzed because a chemical separation procedure precedes the gamma spectroscopy analysis. Paragon will evaluate the technical and economic advantages of using a ^{133}Ba tracer for this analysis.

Item 9) Paragon does not have a formal protocol limiting the total error in tracer measurement to a specific maximum. In practice, the chemists were adding enough tracer activity to adequately limit the associated counting uncertainty. However, we suggest that the laboratory consider implementing a formal quality control criterion to address this issue.

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Response 9) Paragon has revised SOP 714 to formally incorporate a requirement to spike at an appropriate level and count samples long enough to gather 400 tracer counts (thereby limiting the uncertainty associated with the yield correction in alpha spectroscopic measurements). Similarly, Paragon has revised the TPU calculation to quantitatively reflect the counting uncertainty associated with yield corrections in alpha spectroscopy. Please see *Attachment 10* for SOP 714, Revision 5.

Item 10) *Per instruction from LANL, Paragon is calibrating for alpha in GAB analyses using 241Am. Paragon should be aware that 230Th is the required calibrant in gross alpha work for the other DOE-AL facilities. It will be necessary for the laboratory to use 230Th if work is done for Sandia, Pantex, or GJPO. No response is required from Paragon for this item.*

Observation 11:

The following items pertain to organic extract cleanup procedures and the associated logbooks.

Item 1) *Paragon has an SOP for method 3640 (GPC), but review of the run logs showed that the procedure has not been used since February of this year. LANL chemists indicate that there have been no significant problems with surrogate failures or inappropriate dilutions at Paragon. However, we recommend that Paragon negotiate appropriate compensation with LANL and ensure that GPC cleanup is performed as needed.*

Response 1) Paragon has provided a fee schedule to LANL that includes a line item for GPC/Method 3640 cleanup. In order to ensure that organic cleanups are performed as needed, the QA Manager has written and distributed a memorandum regarding the use and application of various organic extract cleanup procedures. The memorandum will be distributed and discussed at an Organics Department meeting on December 02, 1999. Please see *Attachment 11* for a copy of the memorandum.

Item 2) *Paragon does not have an SOP for 3610 (alumina) cleanup.*

Response 2) Paragon has revised SOP 618, "Alumina Column Cleanup -- Method 3610B," and distributed controlled copies of Revision 3. Please see *Attachment 11* for a copy of SOP 618, Revision 3.

Item 3) *Paragon does not have a current approved SOP for 3620 (florisil) cleanup. That SOP was retired, the cleanup appears to generally not be done, and the old SOP was never updated to reflect the use of cartridges as opposed to packing columns.*

Response 3) Paragon has revised SOP 648, "Florisil Cleanup -- Method 3620B," and distributed controlled copies of Revision 2. Please see *Attachment 11* for a copy of SOP 648, Revision 2.

Item 4) *Paragon has an SOP for 3630 (silica gel), but that SOP has not been reviewed since June of 1996.*

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Response 4) Paragon has revised SOP 604, "Silica Gel Cleanup of Polynuclear Aromatic Hydrocarbons Extracts -- Method 3630," and distributed controlled copies of Revision 4. Please see *Attachment 11* for a copy of SOP 604, Revision 4.

Item 5) Paragon does not have an SOP for 3660 (sulfur cleanup). Elemental sulfur interferes with analyte compounds in both 8081 and 8082.

Response 5) Paragon has revised SOP 634, "Sulfur Cleanup -- Method 3660B," and distributed controlled copies of Revision 3. Please see *Attachment 11* for a copy of SOP 634, Revision 3.

Item 6) Paragon has an SOP for 3665 (H2SO4/KMnO4 cleanup). However, that SOP does not discuss the use of KMnO4 or under what conditions that might be needed.

Response 6) Paragon has revised SOP 651, "Sulfuric Acid Cleanup of PCB Extracts -- Method 3665A," and distributed controlled copies of Revision 4. Paragon notes that the sequential use of permanganate is optional and is not performed by our laboratory. Please see *Attachment 11* for a copy of SOP 651, Revision 4. In addition, Paragon has created a Sulfur Cleanup Logbook and Form 645 follows for your review (*Attachment 11*).

Item 7) The extract cleanup notations in some run logs were unclear and did not reference the regulatory method numbers.

Response 7) Paragon has reviewed all extraction forms. Particular focus was given to clean up procedures and regulatory references. Revised forms have been implemented and follow for your review (*Attachment 11*).

Item 8) Several entries in an 8270 extraction log had been obliterated, and the GPC run log did not provide all the information required by section 3.2.1 of the Model SOW. In general, the applicable SOP number had not been entered in the corresponding fields of extraction logs.

Response 8) Although the auditor's comment pertains to the Extractions Group, a labwide re-training session was conducted for all preparatory personnel and supervisors. This training session highlighted: good laboratory documentation practices; completeness of benchsheets (particularly SOP/REV documentation); and enhancing completeness as part of the supervisory review. The training sign-off sheet follows with *Attachment 7* for your review.

Observation 12:

Paragon's sample-login system was well developed and effective. However, based upon our review of completed login documents, we offer several suggested areas for improvement below.

Item 1) COC and sample label legibility is not specifically addressed on the login worksheet.

Response 1) Paragon has revised Form 201 to address the concern of COC and sample label legibility. Please see *Attachment 12* for a copy of Form 201.

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Item 2) The login documents do not distinguish between an airbill that was missing and one that was not removable. When airbill labels are not removable, the shipper's tracking number is not being recorded at present.

Response 2) Paragon has revised Form 201 to distinguish between airbills that are missing and/or not removable. In addition, Paragon has added language to allow for recording of shipper's tracking number. Please see **Attachment 12** for a copy of Form 201.

Item 3) Internal COCs are not included in data packages at present. While not specifically required, Paragon could include these to complete the custody records.

Response 3) It is true that Paragon does not routinely provide internal chain of custody documents in data packages, because clients have not requested these documents as of this writing. Paragon maintains internal chain of custody for **all** work orders and will gladly provide these documents, upon client's request.

Item 4) Samples that are shipped in the same container with broken samples are not listed during login. Doing this might help explain unexpected contamination.

Response 4) Paragon has revised Form 201 to require that specific sample IDs are recorded when broken and intact samples are received in the same cooler. Please see **Attachment 12** for a copy of Form 201.

Item 5) There are some codes used on the login worksheets that are not defined. Paragon should avoid using undefined symbols and codes.

Response 5) Paragon has revised Form 201 to include definitions of codes or to delete codes. Please see **Attachment 12** for a copy of Form 201.

Item 6) The login worksheet lists the acceptable shipment temperature as "< 6°C." The worksheet should be amended to say "between 2°C and 6°C."

Response 6) Paragon has revised Form 201 to address this issue. Please see **Attachment 12** for a copy of Form 201.

Item 7) Reagent grade HNO₃ is used to adjust pH during login when necessary. Paragon should use trace-metals grade acid for this.

Response 7) Paragon concurs that trace-metals grade acid should be used by the Sample Control Department to adjust the pH of aqueous samples and prevent contamination of samples intended for metals analysis. Trace-metals grade nitric acid has been provided to the Sample Control Department and the requirement to use this grade of acid discussed with the Sample Control Custodian, Jeannine Hunter. SOP 202, Login and Distribution of Samples, has been revised to note the requirement of trace-metals grade acid for acid preservation of metals samples. Please see **Attachment 12** for relevant pages from SOP 212, Revision 3.

Item 8) The 16-hour holding time prior to aliquoting samples that have had pH adjustment is specified only for 200.7 metals. Acid preservation in metals analysis is done to

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prevent precipitation and adsorption onto the container walls. Those conditions can arise in any metals sample, and hence we recommend that the hold time after adjustment be extended to all acid preserved samples.

Response 8) Paragon has revised SOP 202 to require that all samples intended for metals analyses be held for 16 hours before analysis, if the pH was adjusted at the laboratory. Please see **Attachment 12** for a copy of SOP 202, Revision 3.

Item 9) *The login SOP lists the acceptable pH for base preserved samples as " ≥ 10 ." This is not adequate for all basic samples; CN requires a pH ≥ 12 .*

Response 9) Paragon has revised SOP 202 to specify required pH ranges, method of measuring pH, and the use of narrow-range pH paper for determining the pH of samples. In addition, Paragon presents an internal memorandum that specifies **acceptable pH ranges for each method**. Please see **Attachment 12** for a copy of SOP 202, Revision 3 and the internal memorandum of November 16, 1999.

Observation 13:

The following remarks apply to various general inorganic analyses.

Item 1) *For ICP-AES work, Paragon has not implemented the GFAA spiking levels specified in the Model SOW. However, the lab has done the development work and routinely provides those levels for other clients.*

Response 1) Paragon has modified spiking levels for LANL analyses in accord with the Model SOW. Spiking levels will emulate the CLP SOW ILMO4.0 for metals by ICP-AES and GFAA, as required by the contract. This change in spiking level was implemented on November 15, 1999.

Item 2) *For ICP-AES work, LANL has apparently requested in the past that analytical spike data not be included in data packages. However, some analytes, notably lead, may be too high in samples for the GFAA spiking levels to yield meaningful data. In those cases, analytical spikes are performed by Paragon's chemists and should be reported. We believe that Paragon and LANL are now in full agreement on items 1 and 2.*

Response 2) Paragon agreed to provide post-spike data in level IV data packages for LANL. This change was implemented on November 15, 1999.

Item 3) *For TOC analyses, there are no daily instrument sensitivity checks at present.*

Response 3) Paragon encloses pages from the TOC logbook that demonstrate daily instrument sensitivity checks. Please see **Attachment 13** for this information.

Item 4) *For TOC work, the chemist is keeping working standards for 6 months. The applicable Paragon SOP limits shelf life to 3 months and the Model SOW limits shelf life to one day for most of his working levels.*

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Response 4) Paragon has revised SOP 300 to correct this error and require that working standards be replaced daily, in accordance with the Model SOW. Please see *Attachment 1* for a copy of SOP 300, Revision 3.

Item 5) For ion chromatography, the QC section of the SOP doesn't require analysis of a final CCB, lacks some QC acceptance criteria, and does not give corrective actions for QC failures in some cases. The QC section of that SOP should be reviewed and revised to comprehensively present QC sample type, frequency, acceptance criteria, and corrective actions for failures.

Response 5) Paragon will revise SOP 1113, "Determination of Inorganic Anions by Ion Chromatography -- Methods E300.0 and SW9056" to address the analysis of a final CCB and include the QA/QC table now required for analytical SOPs. Debra Henderer and Darryl Patrick are responsible for revising this SOP by December 31, 1999.

Item 6) For IC, the chromatography is poor in the vicinity of the fluoride peak. Paragon chemists should add eluent to all standards and samples to smooth the water ditch, use a column that separates the fluoride peak from the water ditch, or both.

Response 6) Paragon has purchased a new ion chromatograph and installed a different analytical column that separates the fluoride peak from the water ditch. Samples chromatograms are enclosed for your review (please see *Attachment 13*).

Item 7) For sulfide analysis, Paragon does not have an approved SOP at present. In addition, the calculations for quantifying unknowns and establishing the normality of the iodine solution are not given on the associated worksheets.

Response 7) Paragon has written an SOP for sulfide analysis. SOP 1120, Revision 0 follows for your review as *Attachment 13*. Paragon will verify that the calculation for quantifying unknowns and establishing normality of the iodine solution include the appropriate equation. Debra Henderer and Darryl Patrick are responsible for ensuring that the worksheet is updated by December 31, 1999.

Thank you again for your time and assistance during the on-site audit. We hope that our responses meet your requirements. Please contact me at 970 490 1511 if additional information is required and I will be glad to provide it.

Respectfully Submitted,

Debra Henderer
Quality Assurance Manager
Paragon Analytics, Inc.

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Enclosures (to Mark Minter only)

cc: Mark Minter (AGRA E&E)
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Via Federal Express

March 06, 2000

U.S. Army Corps of Engineers
ATTN: CENWO-HX-C (Laboratory Validation Coordinator)
12565 West Center Road
Omaha, NE 68144-3869

**RE: *On-Site Inspection of Paragon Analytics, Inc. by
New Technologies Environmental Consulting, Inc.
February 16-18, 2000
Draft Response to Findings***

Dear Laboratory Validation Coordinator:

I am writing to respond to Ms. Rhonda Carter's audit report of February 18, 2000. Paragon sincerely appreciates Ms. Carter's on-site audit of our systems and processes and the time spent with our employees. Paragon's responses, supporting documentation of completed corrective actions, and proposed corrective actions (with implementation schedule and responsible parties named) follow for your review.

General

Item (1) D: *Dilution and mixtures of reagents, solvents, and calibration standards are not consistently traceable.*

Response (1) D: Paragon concurs that all standards, solvents, reagents, preservatives, dilutions, mixtures, and drying salts must be traceable to the source and that this traceability shall be documented by a unique identifier number on all preparatory benchsheets and/or analytical run sequence logs as appropriate. The QA Department has addressed this finding by: (1) providing a laboratory-wide training on the topic of unique identification and traceability; (2) editing preparatory benchsheets and analytical run sequence logs; and (3) replacing bound logbooks with updated forms. Copies of the signed laboratory-wide training form and representative preparatory benchsheets and



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analytical run sequence logs follow for your review (please see *Attachment General (1) D*).

Item (2) D: *The laboratory allows the use of uncontrolled documents ("cheat sheets") printed from the LIMS system as a reference for control limits of the LCS, MS/MSD, and surrogates.*

Response (2) D: Paragon acknowledges that, prior to the USACE audit, we allowed analysts to post "cheat sheets" for their reference. Paragon notes that control limits for LCS, MS/MSD, surrogates, and chemical tracers are electronically controlled by the QA Department through our Laboratory Information Management System (LIMS). Therefore, evaluation and reporting of data are always performed against updated, controlled limits and not the "cheat sheets" that analysts used.

Per the auditor's request, the QA Department has instructed that all "cheat sheets" be eliminated and that only controlled documents may be used as a reference. On February 28, 2000, the QA Department verified that all uncontrolled documents had been removed from the laboratory. In addition, the QA Department conducted a laboratory-wide training on this topic. Copies of the laboratory-wide training form follow for your review (please see *Attachment General (2) D*).

Item (3) R: *The reporting limit for methods should be at least 3-5 times the MDL.*

Response (3) R: Historically, Paragon has followed the AFCEE 3.0 QAP guidance, which allows an MQL value no lower than two times (2x) the MDL. On February 24, 2000, Paragon obtained the latest version of the USACE Shell for Analytical Chemistry from Dr. Richard Kissinger. Paragon agrees to follow Section 3.3.7.2 of this document, which states that the method quantitation limit (MQL) values shall be no lower than three (3x) times the MDL for any target analyte. The QA Department provides special instructions to every laboratory for MDL studies and a copy of the revised special instructions follow for your review (please see *Attachment General (3) R*). Paragon respectfully notes that following repeated attempts to comply with prescribed criteria, the QA Department may tolerate marginal failures for a long list of compounds (e.g., Methods 8260B, 8270C).



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Sample Receipt

Item (1) D: *The IR thermometer gun is calibrated annually.*

Response (1) D: Historically, Paragon has contracted annual calibration and certification of the IR thermometer to RHQ Calibration Facilities. Per USACE Shell requirements stated in Section 9.1.2, Paragon will supplement the annual calibration and certification with internal, quarterly verifications. Paragon has prepared an SOP 938, "Verification of the IR Temperature Gun," which addresses the internal, quarterly, two-point verification of the IR thermometer. The first quarterly verification of the IR temperature thermometer was performed in-house on March 03, 2000. Paragon submits recent vendor certificates of calibration, SOP 938, completed Form 304, and documentation of scheduled quarterly verifications for the months of March, June, September, and December for your review (please see *Attachment Sample Receipt (1) D*).

Item (2) D: *The correction factor is not taken into account when reading and recording temperature.*

Response (2) D: Paragon performs annual verifications on all thermometers against a thermometer certified traceable to NIST. Per the auditor's request, Paragon notes that the QA Department has tightened the acceptable tolerance range -- based on the capability of the thermometer -- in order to preclude the application of a correction factor when recording the observed temperature. For example, narrow-range thermometers that are dedicated to specific applications, such as measuring the temperature of refrigeration and freezer units, are readable to the nearest tenth of a degree Celsius (± 0.1 °C) and Paragon has established a new, tighter tolerance range of ± 0.4 °C for these types of thermometers. Wide-range thermometers that are designed for general use applications such as measuring the ambient temperature or a hot water bath's temperature are readable to the nearest degree Celsius (± 1 °C) and Paragon has established a tolerance range of ± 1 % of the total range or ± 1 °C, whichever is less, for these types of thermometers. It is Paragon's understanding that the application of these more stringent tolerances will preclude the need to apply correction factors when recording daily readings.

Thermometers are scheduled to be re-verified in March 2000, according to the procedures set forth in revised SOP 923. Paragon will forward completed verifications for your review by March 31, 2000. SOP 923, which sets forth the criteria described above, follows for your review (please see *Attachment Sample Receipt (2) D*).



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Trace Metals Preparation Areas

Item (1) D: *The calibration frequency of the in-line conductivity (sic) meter is unknown.*

Response (1) D: Per the requirements of our LANL DOE contract, Paragon purchased an in-line resistivity meter from Thornton Associates in July 1997 (which must be used instead of a conductivity meter). According to Thornton Associates, the in-line resistivity meter need not be re-calibrated under conditions of normal use. The certificate provided by the manufacturer, including documentation of acceptable calibration upon installation, follows for your review (please see *Attachment Trace Metals Preparation Areas (1) D*).

Paragon believes that our system consistently produces finished water that meets ASTM's *strictest standards for Type I water* (ASTM 1193, "Standard Specification for Reagent Water," Type I specification: minimum 16.67 MΩ-cm = maximum 0.06 μmho/cm for the following reasons.

- (1) Paragon's primary deionized water system is maintained, under contract, by U.S. Filter Corporation, who routinely evaluates and replaces the tanks. The primary system consists of the following tanks in series: two (2) 2.1 ft³ mixed bed tanks; one (1) 1.2 ft³ carbon filter tank, one (1) 2.1 ft³ cation exchange tank, one (1) 2.1 ft³ anion exchange tank (please see *Attachment Trace Metals Preparation Areas (1) D*).
- (2) Finished water from Paragon's primary deionized water system passes through a *secondary* Milli-Q system and its in-line resistivity meter before being dispensed to the laboratories. The secondary treatment system is also designed to produce finished water that meets Type I specifications.
- (3) Evaluation of reagent and instrument blanks for all organics, metals, and wet chemistry parameters indicates that the two (2) independent, in-series water systems consistently produce finished water that yields reagent blanks without contaminants at the level of detection.

In summary, Paragon believes that its approach to producing and monitoring high purity water is equivalent to the one outlined in the USACE Shell for Analytical Chemistry, Section 9.1.4. Paragon respectfully requests your consideration of our approach.



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Item (2) D: *The conductivity of the reagent water is not checked and documented daily. Currently the lab is recording the meter reading daily.*

Response (2) D: Paragon's two (2) in-line meters measure resistivity (in $M\Omega\text{-cm}$), which is the reciprocal of conductivity (in $\mu\text{mho/cm}$). Paragon requires daily verification and documentation of the two (2) independent, in-series systems. Representative logbook pages from the systems recordings follow for your review (please see **Attachment Trace Metals Preparation Areas (2) D**). Please note that both systems consistently produce water that exceeds Type I specifications (minimum $16.67 M\Omega\text{-cm}$ = maximum $0.06 \mu\text{mho/cm}$). As stated in the previous response, Paragon believes that its approach to producing and monitoring high purity water is equivalent to the one outlined in the USACE Shell for Analytical Chemistry, page 27, Section 9.1.4. Paragon respectfully requests your consideration of our approach.

Metals via ICP (Method 6010B)

Item (1) D: *The post digestion spike is only run when the MS/MSD fails.*

Response (1) D: Section 10.3.1 of the USACE Shell for Analytical Chemistry describes incorporating post digestion spikes (PDS) into an analytical sequence to assess matrix effects based upon: (1) the occurrence of new and unusual matrices or (2) contingency analysis based upon serial dilution (SD) or matrix spike (MS) failures. Paragon performs matrix spikes and serial dilutions for every batch and for any new or unusual matrices (e.g., concrete, oil, wipes) in order to evaluate matrix effects. Historically, Paragon has performed PDS only for all analytes that fail the matrix spike recovery criteria. Following our reading of the Shell, Paragon agrees to follow Section 10.3.1 for USACE contracts by performing a PDS for serial dilution failures (in addition to matrix spike failures).

Item (2) D: *The lowest point on the calibration curve is greater than the reporting limit.*

Response (2) D: Paragon will change its ICP-AES calibration practices to comply with calibration requirements for Method 6010 as described in Section 9.2.1.1 of the Shell. We will follow calibration option 2 (three standards plus a calibration blank, linearity evaluation, low level calibration standard at the reporting limit). SOPs 807 and 805 will be revised to include this low-level standard, followed by a departmental training



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session. Paragon will forward revised SOPs 807 and 805; sample analytical run logs; and documentation of training by March 31, 2000.

Item (3) D: *The analyst was unfamiliar with the initial calibration curve acceptance criteria. The analyst used the check standard to evaluate the initial calibration curve.*

Response (3) D: All ICP analysts and data reviewers will be retrained on Method 6010B requirements, Paragon (revised) SOP 807 and 805 requirements, and the USACE Shell requirements. Paragon will forward documentation of the training by March 31, 2000 (please see previous response for related SOP revision and training discussion).

Item (4) D: *The MDLs for several analytes do not meet the 10X spiking criterion.*

Response (4) D: For the most recent MDL study for Method 6010B, the analytes that did not meet the 10X spiking criterion are the following cations: aluminum, calcium, iron, magnesium, and sodium. The spiking level for these cations was 0.1 mg/L, which is at or below the reporting limit. Calculated MDL values were less than 0.01 mg/L (10X rule failure). In theory, respiking at a lower level should result in meeting the 10X spiking criteria and in calculating the "real" MDL value. As of this writing, Paragon has accepted this MDL study for the following reasons:

- (1) The native concentrations of these low-toxicity metals are usually relatively high for field samples. Multi-point calibrations covering a range from as high as 500 mg/L to the reporting limit are performed for these elements. Because the calibration method does not include multiple standards near the origin, accuracy at concentrations significantly below the reporting limit is not known. Although high precision and large signal/noise ratios of an ICP yield MDLs in the low ppb range, it may not be possible to accurately quantitate in the 0.01-0.1 mg/L range because of the calibration approach.
- (2) It is possible that the calculated MDLs for these analytes are higher than the real MDLs, as the data were acquired using solutions with analyte concentrations greater than 10x the calculated MDL. If the MDL studies were repeated at lower concentrations, the variances would also be lower, which would result in a lower MDL value. Although the calculated MDLs may be higher than the real MDLs, they are well below the reporting limit and need not be any lower for our applications.



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Paragon respectfully requests further technical discussion on this issue for the cations reported by Method 6010B.

Metals via Graphite Furnace (Methods 7000 series)

Item (1) O: *The laboratory uses this method very little and prefers to use the Trace ICP.*

Response (1) O: Paragon has only one (1) graphite furnace instrument; therefore, analysis of multiple metals is impractical for a production laboratory. We prefer to perform all metals analyses by ICP-AES radial or axial analyses, as appropriate. Paragon's Sales Representatives discuss substitution of graphite furnace methods at the time of procurement.

Mercury (Methods 7470/7471A)

Item (1) D: *The laboratory deviates from Method 7471. The sample is not heated for 2 minutes at 95 °C following the addition of aqua regia.*

Response (1) D: SW-846 Method 7471, Section 7.1 and Paragon's revised SOP 812, Revision 6, Section 8.2.6 clearly state the requirement to heat the sample for 2 minutes at 95 °C following the addition of aqua regia. Paragon has re-trained all metals digestion analysts to ensure compliance with the method and SOP requirements. Copies of the training form follow for your review (please see *Attachment Mercury (1) D*).

Volatiles Support Areas

Item (1) D: *The analytical batch is not defined by the loading of the purge unit for Method 8015 GRO and 8021B.*

Response (1) D: Paragon understands that if the laboratory does not have a closed-system purge and trap unit (e.g., Archon® Purge and Trap Autosampler), then the analyst must load a method blank after every sample if he/she steps away from the instrument or, alternatively, the analyst must include a "room" blank at the end of the sequence in order



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to monitor laboratory contamination that may occur during the loading process. Paragon will comply with this USACE requirement. We will submit revised SOPs and documentation of re-training for all GC Fuels analysts by March 31, 2000.

Item (2) D: *The stock gas and intermediate standards do not expire in a timely manner. Standards expire in 1 month.*

Response (2) D: Paragon understands that this finding relates to opened stock and intermediate standards for GC/MS and GC volatiles gas standards (i.e., those compounds whose boiling point is less than 30 °C). At the time of the audit, Paragon's SOP 300, Revision 6 prescribed a one (1) month expiration date for opened stock and intermediate gas standards. Per the auditor's request, Paragon has instituted a shorter expiration date of one (1) week for opened stock and intermediate standards. Paragon submits SOP 300, Revision 7 for your review (please see *Attachment Volatiles Support Areas (2) D*).

Volatiles via GC/MS (Method 8260B)

Item (1) D: *The MDLs for the 5 mL purge was not available during the audit.*

Response (1) D: The MDL studies performed in January and February for Method 8260B, 5 mL purge, for all three (3) instruments have been evaluated by the QA Department and determined to be unacceptable with respect to the 10X spiking/MDL criterion and the 3X MDL/RL criteria. New MDL studies are being performed and will be submitted for your review by March 31, 2000. The January and February summaries follow for your review (please see *Attachment Volatiles via GC/MS Method 8260B (1) D*).

Item (2) R: *The drift criterion for the CCV should be applied to all analytes.*

Response (2) R: In general, Paragon follows the guidance in Method 8260B, Section 7.4.5.2. Paragon agrees to comply with the guidance stated in the Shell, Section 9.5.2.4 as required for USACE projects (i.e., evaluation of the CCCs and all project-specified contaminants of concern at $\pm 20\%$ of expected value.)

Item (3) R: *The grand mean (an average of all of the RF averages) should not be used for evaluating the linearity of the initial calibration curve.*



Response (3) R: Paragon understands that the following documents prescribe the use of the grand mean for evaluating linearity: Method 8000B, Section 7.5; Method 8260B, Section 7.3.8; USACE Shell, Section 9.2.2.4 and Table 12. Paragon's practices are compliant with these documents. Paragon respectfully invites further discussion of this item.

Item (4) O: *TICs are reported upon request.*

Item (5) O: *Method 5035 is used to prepare low-level soil samples.*

Volatiles via GC (Method 8021B)

Item (1) D: *Low-level soil samples cannot be prepared using Method 5035. The laboratory encourages clients to use Method 8260.*

Response (1) D: Paragon does not have a closed system purge and trap autosampler, which is required to perform Method 5035, for the GC Volatiles laboratory. Paragon performs Method 5030A for GC Volatiles analytical methods. Paragon discloses the preparatory method in its price list and in all proposals. Paragon's decision not to perform Method 5035 in the GC Volatiles laboratory is based on the cost of an Archon® Autosampler and the lack of request for Method 5035 in this area.

Item (2) O: *The lab only runs an abbreviated analyte list. The GCs do not have Hall detectors. The instruments are currently in the process of being upgraded. New detectors are being installed.*

Response (2) O: In performing Method 8021B, Paragon calibrates for the truncated list of compounds that are can be detected by a PID and confirms by a dissimilar analytical column. Paragon calibrates for and reports the following compounds by Method 8021B: benzene, toluene, ethylbenzene, xylenes, chlorobenzene, and dichlorobenzenes.

Per the auditor's request, Paragon will submit new retention time (RT) window studies, MDL studies, and IPRs for Method 8021B. Paragon will submit this documentation by March 31, 2000.



TPH GRO/DRO (Method 8015M)

Item (1) D: *The SOPs for GROs and DROs are in draft form.*

Response (1) D: The SOPs for GRO and DRO analyses have been in existence since 1992 and were being revised at the time of the audit. Paragon encloses SOP 406, Revision 6 (GRO) and SOP 425, Revision 6 (DRO) for your review. Please see **Attachment TPH GRO/DRO (1) D).**

Prior to the on-site audit, Paragon submitted Revision 5 of the GRO and DRO SOPs to the Laboratory Validation Coordinator. In response to the USACE reviewer's comments, Paragon notes that LCS qc limits are not listed in our SOPs, as these limits vary from project to project and are subject to update that may not coincide with the SOP update. QC limits are controlled by the QA Department and updated through our LIMS system. Therefore, evaluation and reporting of data are always performed against updated, controlled qc limits.

Item (2) D: *Low level soils samples for Method 8015M cannot be prepared using Method 5035.*

Response (2) D: Paragon does not have a closed system purge and trap autosampler, which is required to perform Method 5035, for the GC Volatiles laboratory. Paragon performs Method 5030A for GC Volatiles analytical methods. Paragon discloses the preparatory method in its price list and on all proposals. Paragon's decision not to perform Method 5035 in the GC Volatiles laboratory is based on the cost of an Archon® Autosampler and the lack of request for Method 5035 in this area.

Item (3) D: *The MDLs for DROs were not available during the audit.*

Response (3) D: The QA Department has evaluated and approved the DRO MDL studies performed in January 2000. Paragon encloses the solid and aqueous DRO MDL studies for your review (please see **Attachment TPH GRO/DRO (3) D).**

Item (4) O: *The instruments used for GROs are currently in the process of being upgraded; new detectors are being installed.*



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Response (4) O: Per the auditor's request, Paragon will submit new retention time (RT) window studies, MDL studies, and IPRs for Method 8021B. Paragon will submit this documentation by March 31, 2000.

Organic Preparation

Item (1) R: *The clean up procedures SOPs should be referenced in the preparation SOPs.*

Response (1) R: Paragon will add language regarding applicable clean up procedures to the following sections of the extraction SOPs: Section 1, Scope and Application and Section 2, Method Summary.

<u>Analytical SOP</u>	<u>SW-846 Reference</u>
SOP 617	Method 3520B
SOP 625	Method 3540C
SOP 626	Method 3510C
SOP 647	Method 3550C

The information added to the SOPs will be taken from the training session recently given by the QA Department. Please see the attached documentation for a summary of the training session (please see *Attachment Organic Preparation (1) R*).

Explosives (Method 8330)

Item (1) D: *The MDLs for several analytes do not meet the 10x spiking criterion.*

Response (1) D: Paragon is performing new MDL studies for Method 8330 in order to achieve the 10X criteria. We will submit summaries of the MDL studies by March 31, 2000.



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Pesticides/PCBs/Herbicides (Methods 8081A/8082/8151A)

Item (1) D: *There is no backup analyst in the area.*

Response (1) D: Ms. Lori Elshof is the backup analyst for the gc semivolatiles area and she was being trained in various gc semivolatiles analyses at the time of the audit. Paragon submits documentation of completed training for your review. Please see **Attachment Pesticides/PCBs/ Herbicides (1) D** for copies of Ms. Elshof's IPRs and SOP/Method Review forms.

Item (2) D: *The columns were replaced in all instruments The retention time window studies and MDLs were not available.*

Response (2) D: Prior to the audit, the columns in two (2) instruments, ECD3 and ECD4 were replaced. Paragon submits new RT window studies for your review. Please see **Attachment Pesticides/PCBs/Herbicides (2) D** for copies of these RT window studies.

New MDL studies are being evaluated as of this writing. Paragon will submit new summaries by March 31, 2000.

PAHs (Method 8310)

There are no additional findings in this area.

Semivolatiles via GC/MS (Method 8270C)

Item (1) R: *The grand mean (an average of all of the RF averages) should not be used for evaluating the linearity of the initial calibration curve.*

Response (1) R: Paragon understands that the following documents prescribe the use of the grand mean for evaluating linearity: Method 8000B, Section 7.5; Method 8270C, Section 7.37.1; USACE Shell, Section 9.2.2.4 and Table 12. Paragon's practices are compliant with these documents. Paragon respectfully invites further discussion of this topic.



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Item (2) R: *The lab should apply the drift criterion for the CCV to all compounds.*

Response (2) R: In general, Paragon follows the guidance in Method 8270C, Section 7.3.5.4. Paragon agrees to comply with the guidance stated in the Shell, Section 9.5.2.4 as required for USACE projects (i.e., evaluation of the CCCs and all project-specified contaminants of concern at $\pm 20\%$ of expected value.)

(3) O: *TICs are reported upon request.*

Anions (Methods 300 series / 9056)

Item (1) D: *Unauthorized undated changes were made to the SOP.*

Response (1) D: On February 28, 2000, the QA Department verified that all uncontrolled documents had been removed from the laboratory. In addition, the QA Department conducted a laboratory-wide training on this topic. Copies of the laboratory-wide training form follow for your review (please see **Attachment General (2) D**).

Item (2) D: *The stock standards and dilutions expire in 1 year.*

Response (2) D: Paragon has rewritten SOP 300 to comply with the standard expiration dates given in Method 9056, Section 5 (see, in particular, Sections 5.5.5 and 5.7). Paragon submits SOP 300, Revision 7 for your review (please see **Attachment Volatiles Support Areas (2) D**).

Item (3) D: *The working calibration curve is run daily upon request.*

Response (3) D: Paragon's SOP 1113 and routine practice complies with Method 9056, Section 7 calibration criteria, which does not require daily recalibration if the opening CCV standard passes retention time and response criteria ($\pm 10\%$). However, Paragon does perform daily recalibration in order to comply with project specific requirements. Paragon is unable to find the requirement to perform daily calibration and requests your assistance in locating the written requirement.



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Item (4) D: *The MDL for nitrite (sic) does not meet the 10X spiking criterion.*

Response (4) D: Paragon acknowledges that nitrate failed the 10X spiking criterion for the aqueous MDL study. Nitrate was spiked at 40 ug/L and the MDL calculated at 3.77 ug/L. Paragon will perform a new aqueous MDL study for Method 9056 and submit a summary by March 31, 2000.

Cyanide (Method 9010A)

Item (1) D: *The high and low distilled standards are not performed.*

Response (1) D: Paragon has revised SOP 1110 and revised our practice to include the requirement of high and low distilled standards. Paragon submits SOP 1110, Revision 2 for your review (Please see *Attachment Cyanide (1) D* for the revised SOP). See Section 8.3.2.1 for this addition.

Item (2) D: *The tolerance of the columns used to determine initial and final volumes are unknown.*

Response (2) D: Per the auditor's direction, Paragon has performed an experiment to determine the accuracy of the midi-distillation tubes' volume markings. We selected 20 random tubes and determined the initial weight of each. We then filled each tube with 50 mL of DI water and reweighed the tube and its contents (assumption: 50.0 g = 50.0 mL). This procedure was repeated two (2) times. The data presented in *Attachment Cyanide (2) D* demonstrate that the greatest absolute deviation is 1.6%.

Item (3) D: *The variable volume pipettes are not checked daily at point of use.*

Response (3) D: Paragon believes that this finding is the result of a misunderstanding. Paragon does perform daily verification of all pipettes before use. We submit copies of logbook pages and representative cyanide benchsheets that demonstrate daily verification (please see *Attachment Cyanide (3) D*).

Item (4) D: *The 1000 uL KCN standard solution prepared in-house is not standardized.*



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Response (4) D: Paragon has ordered the required standard in order to comply with this request. We enclose a copy of a purchase order for silver nitrate (please see *Attachment Cyanide (4) D*). Following receipt of this standard, we will standardize the 1000 uL KCN standard solution.

Item (5) R: *The photometric standard used to verify wavelength and absorption should be performed quarterly.*

Response (5) R: Paragon has ordered a Wavelength Standard from Barnstead-Thermolyne in order to perform the quarterly verification of wavelength and absorption. Following receipt of this standard, we will establish a quarterly schedule. Please see *Attachment Cyanide (5) R* for a copy of the purchase order for the Wavelength Standard.

TRPH/Oil & Grease (Methods 418.1/413.2)

Item (1) D: *The temperature of the samples is not specified when determining sample aliquot.*

Response (1) D: Paragon understands this finding to relate to the temperature/density relationship that may be important when weighing out aqueous samples. The enclosed table from the CRC "Handbook of Chemistry and Physics" demonstrates that density changes with temperature. The difference in the density of water at Paragon's typical ambient temperatures (18-25 °C) is 0.00155 g/mL, which is negligible in comparison to the 1.0% tolerance applied in the volumetric calibration procedures. Note that the total deviation from a maximum density of 1.00000 g/mL (at 3.98 °C) is 0.00293 g/mL, which is still a negligible amount. (Please see *Attachment Cyanide (1) D*).

Further, Paragon reports a maximum of three (3) significant figures; therefore, volumetric inaccuracies in the ranges described above are undetected.

Given the facts presented above, Paragon believes that recording temperatures and performing density adjustments of aqueous samples is unnecessary. We respectfully invite further discussion of this topic.

Item (2) D: *IR spectrophotometric accuracy and repeatability is not checked and documented using NIST-traceable standards.*



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Response (2) D: Paragon uses a Buck Scientific Model HC-404 to perform Methods 418.1 and 413.2. This instrument is a single wavelength spectrophotometer that is designed for quantitative measurement of hydrocarbons dissolved in a non-absorbing solvent such as freon. The analytical wavelength is isolated using a narrow band infrared filter (2924 cm^{-1}). No other analytical wavelength is available. Please see *Attachment TRPH/Oil & Grease (2) D* for information from the manufacturer.

We are not aware of a procedure that can be used to verify the accuracy of the wavelength. As the instrument does not have a scanning capability and the use of an optical filter ensures selection of the proper wavelength, Paragon does not believe the suggested check is necessary. We respectfully invite further discussion of this topic.

Item (3) D: *The low standard is higher than the reporting limit and the reporting limit is not verified.*

Response (3) D: Paragon believes that this finding is the result of a misunderstanding. The lowest calibration standard is lower than the reporting limit. The source of the confusion is not considering the concentration factor (10X) that occurs during the extraction process. The initial water sample volume is 10x times the final freon extract volume and the correction factor must be considered in comparing standard levels to the reporting limit. The analytical curve consists of standards at the following instrument levels: 0, 5, 10, 50, 100, and 500 mg/L in freon (which equate to 0.5, 1, 5, 10, and 50 mg/L reported value). Paragon's level 2 standard is 10 mg/L, which equates to the reporting limit of 1 mg/L. Paragon's level 1 standard is 5 mg/L, which is lower than our established reporting limit of 1 mg/L and equates to a reporting limit of 0.5 mg/L. Representative logbook pages and preparatory sheets follow for your review (please see *Attachment TRPH/Oil & Grease (3) D*).

TOC (Method 9060)

Item (1) D: The SOP is in draft form.

Response (1) D: The TOC SOP has been in existence since 1994 and was being revised as the time of the audit. Paragon encloses SOP 803, Revision 4 for your review (please see *Attachment TOC (1) D*).



Data Reporting and Review

Item (1) R: *The first tier review used for the inorganics area should include LCS/ MS/MSD and blanks. The first tier (analyst) review should be conducted in wet chemistry area and documented with the use of a checklist.*

Response (1) R: Paragon will modify its wet chemistry and metals checklists per the auditor's request. We will submit revised checklists by March 31, 2000.

Quality Assurance

Item (1) D: The Project Coordination SOP was not available during the audit.

Response (1) D: Paragon has completed SOP 212, which describes project management functions. The SOP is enclosed for your review (please see *Attachment Quality Assurance (1) D*).

Item (2) D: The training files are not adequately maintained for each employee. The analyses of replicates LCSs are not consistently documented. The blind PE sample results are not contained in files.

Response (2) D: Paragon acknowledges that the training files are incomplete. The QA Department will manage the completion of IPRs and annotation of PE performance for each employee by March 31, 2000. Paragon will submit representative files upon request.

Item (3) R: The training content for new employees should include the following: Lab Notebook Control; Data Reduction and Review; Sample Custody, Storage and Disposal; Nonconformance and Corrective Actions; Records Storage and Tracking; Control Charts; Significant Figures; and Laboratory Security.

Response (3) R: As stated above, Paragon acknowledges that the training files are incomplete. The QA Department will manage the inclusion of the items for each employee by March 31, 2000. Paragon will submit representative files upon request.



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Item (4) R: The QA Department should document that analysts have read the QA Manual and its annual updates.

Response (4) R: The QA Department will ensure that all analysts have read the QA Manual and document this training by March 31, 2000. Paragon will submit representative files upon request.

Item (5) R: The signatures list for analysts should be periodically updated.

Response (5) R: The QA Department will update the signature list periodically. Paragon encloses a recent update for your review (please see **Attachment QA (5) R**).

Paragon extends our thanks for your time and consideration of the proposed corrective actions. As stated above, it is Paragon's intent to supply documentation of all corrective actions by March 31, 2000. Please contact me at 970 490 1511 if you have any questions or need additional information.

Respectfully Submitted,

Debra Henderer
Quality Assurance Manager
Paragon Analytics, Inc.

Enclosures

cc: Ms. Rhonda Carter, New Technologies Environmental Consulting, Inc.

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March 15, 1999

Mr. Thomas S. Davis
Quality By Design
97 Puhili Street
Hilo, HI 96720

**RE: On-Site Audit of Paragon Analytics, Inc. on October 26-27, 1998
On Behalf of The Naval Facilities Engineering Service Center (NFESC)
Closure Report**

Dear Mr. Davis:

Thank you for your audit report of November 19, 1998. Paragon Analytics, Inc. (Paragon) appreciates this opportunity to respond to your on-site audit findings and we hope that our responses meet your requirements. Paragon's corrective actions and documentation follow for your review.

4.2 Critical Findings

Item 4.2.1 In the GC/MS volatiles laboratory, several findings were noted:

Item 4.2.1.1 The analysts in this laboratory could not tell the assessor the last date that the mass spectra library for EPA Method 8260 had been updated. Each method's spectral library should be updated on a regular basis.

Reply 4.2.1.1 Paragon's mass spectra TIC library is provided by Hewlett Packard. The library is part of the Enviroquant software package that operates the mass spectrometers. According to Hewlett Packard representatives, the TIC library that Paragon is using is the most current one available (NBS 98k).

Paragon concurs that the "daily" or "method" library must be updated regularly against standards. Please see *Attachment A* for the related policy memo and verification of updates. As of this writing, Paragon's QA Manager has verified that all six (6) instruments' libraries have been updated in 1999.



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Item 4.2.1.2 Laboratory practices are inconsistent with EPA Method 8260. EPA Method 8260 describes the initial calibration using an average response factor (RF), and calculating the Relative Standard Deviation (RSD) as a quality control check for the acceptability of the average RF. This method further states that a linear regression may be used for the initial calibration as either the routine calibration procedure or as a fallback if the RSD is out of criteria, providing that the correlation coefficient is acceptable. The laboratory is using the average RF for the initial calibration for all compounds. If the RSD is unacceptable but the correlation coefficient is acceptable, the analyst accepts the average RF and does not change the calculation process to a linear regression.

Reply 4.2.1.2 Paragon believes that our practices are compliant with Methods 8000B and 8260B. Paragon always uses the average RF of all compounds to calculate results because Paragon's GC/MS software is not capable of linear regression calculation. Therefore, Paragon's five-point curve is constructed to ensure that the mean %RSD is less than 15%, in order to ensure compliance with Methods 8260B (Section 7.3.8) and 8000B (Section 7.5). Inspection of several volatile and semivolatile curves reveals that the "typical" average RF is 8-10%. This value is printed at the bottom of Form VI, which is included in Level III/IV packages. Please see **Attachment B** for %RSD calculations from various instruments that demonstrate Paragon's compliance.

Paragon notes that evaluation of correlation coefficients is allowed by AFCEE Handbook 3.0 and is performed for AFCEE SDGs only. Analysts receive work lists and program specifications for all programs; therefore, analysts are aware of qa/qc requirements such as AFCEE, USACE, NFESC. Please see **Attachment B** for typical work lists and review checklists that note qa/qc requirements (e.g., CLP, 524.2, AFCEE, 8260B).

Item 4.2.1.3 Matrix spikes were not analyzed for EPA Method 8260 on October 5, 7, and 9 1998. The laboratory analyzed matrix spikes every 20 samples, and not with every analytical batch, even if the batch contains less than 20 samples.

Reply 4.2.1.3 Paragon understands SW-846 to define a "batch" as a group of 20 or fewer field samples (plus their associated qc samples) that are



processed continuously. For Method 8260B, Paragon recognizes an additional restriction imposed by the 12 hour tune. Each batch requires its own pair of MS/MSD samples. Paragon performs an MS/MSD for each batch if adequate sample volume is provided by the client. If additional sample volume is not available, then Paragon reports a blank spike and blank spike duplicate (full spike list) in order to provide additional information to the client. The concept of a "batch" was discussed with the GC/MS volatiles analysts during a training session conducted on November 20, 1998. Please see *Attachment C* for documentation of this training.

Paragon notes that during program specification, Paragon's Project Manager discusses SW-846 MS/MSD requirements (per batch) with each client and requests that the client provide adequate sample volume and indicate the MS/MSD sample on the chain of custody.

Item 4.2.1.4 The internal standard area counts were not documented as being checked in each sample to determine if they were within a factor of two (50% - 200%) of the area counts of the continuing calibration check.

Reply 4.2.1.4 Paragon's analysts check the internal standard (IS) area count for each field and qc sample against the calibration standard as required by SW-846 and the CLP SOW. Documentation of these IS checks appears in four (4) places: (1) on each quantitation report as a check "✓" mark beside each IS compound; (2) on Form VIII, which is included with Level III/IV packages; (3) on the runlog, by exception/failure only; and (4) on the review checklists. Please see *Attachment D* for documentation of these practices.

Reply 4.2.1 Please note that the Quality Assurance (QA) Manager held training sessions with the Organics Manager, analysts, data reviewers, and data reporters on Methods 8260B and 8000B on 11/06, 11/19, and 11/20. The four items noted above were discussed in detail. Please see *Attachment C* for documentation of these three (3) training sessions.



Item 4.2.2 Both the inorganics and the organics laboratories performed balance checks using a common set of weights that were not certified. The organics extraction laboratory had a second set of uncertified weights that were occasionally used to check balances.

*Reply 4.2.2 Paragon acknowledges that our Class S weights require re-certification. Paragon's QA Plan and lower-tier SOPs state that Class S weights shall be verified by a qualified, independent vendor every 12 months. QA files indicate that the last verification/calibration occurred 05/02/97. The QA Department sent Paragon's set of 21 Class S weights to Denver Instrument Company for re-certification and a copy of the certificate is included for your review. Please see **Attachment E** for this document.*

4.3 Major Findings

Item 4.3.1 In the Organic Analysis Preparation Laboratory, two findings were noted:

Item 4.3.1.1 The analyst stated that Ottawa Sand (or some other solid matrix) was used for the preparation of a matrix blank for soil/solids extraction only if the client requests it. The routine method blank for soil consisted of sodium sulfate only. Method blanks should be on a non-contaminated material of a similar matrix to the samples.

*Item 4.3.1.1 Paragon understands that, per SW-846 protocol, the method blank and blank spike(s) shall emulate the matrix. Paragon's QA Manager discussed this requirement with all organic extractions analysts, instrument analysts, Chromatography Supervisor, and Organics Manager on November 30. The practice of using sodium sulfate as a solid matrix substitute is recognized to be non-compliant and has been discontinued. Paragon now adds Ottawa Sand to all method blanks and blank spike(s) (e.g., Method 8260, 8151, 8270, 8082, 8081, 8141, 8015M). Please see **Attachment F** for a copy of the memorandum that addresses this topic.*

Item 4.3.1.2 The analyst stated that when performing the Diazomethane derivatization step in EPA method 8151 the Chorophenoxy Herbicides, the excess Diazomethane is not destroyed using



Silicic acid. Instead the laboratory relies on the Nitrogen blowdown to remove the excess Diazomethane.

Reply 4.3.1.2 Paragon generates diazomethane by the diazald kit method. We believe our practice to be compliant with Method 8151A, Section 7.5.1.2.2 which allows solvent evaporation. Method 8151A states: "Reduce the sample volume to approximately 2 mL to remove excess diazomethane by allowing the solvent to evaporate spontaneously at room temperature. Alternatively, 10 mg of silicic acid can be added to destroy the excess diazomethane." No corrective action is proposed for this item as Paragon's practices are compliant with Method 8151A.

Item 4.3.2 Several deficiencies were noted in the performance of the TCLP extraction:

Item 4.3.2.1 The preliminary tests of the TCLP extract are not being performed or the documentation does not completely document the percent solids determination (EPA 1311, Sections 7.1.1 and 7.1.2) and the particle size reduction/surface area determination (Section 7.1.3).

Reply 4.3.2.1 Paragon has addressed these requirements. Please see **Attachment G** for copies of the revised TCLP forms and SOPs.

Item 4.3.2.2 The rotation was monitored for the TCLP extraction but the temperature was not. Extraction temperature must be controlled to 23 ± 2 °C and logging must include these extraction conditions.

Reply 4.3.2.2 Paragon has begun monitoring the temperature of the TCLP room on a daily basis. Please see **Attachment H** for a copy of recent log book pages that substantiate our monitoring of the temperature.

Item 4.3.2.3 Multiphasic samples were improperly processed. When the analyst was asked about the handling of samples with two or more phases, such as oil and water or soil, oil, and water, logbook entries for Samples No. 98-09-074-02, -06, and -10 and -14 were reviewed. It appeared as if the total volume of the sample was adjusted for the percent solids determined in the preliminary tests. Typically, approximately 100 grams of sample is extracted. However, 187.6 grams of one sample was extracted due to the level of percent solids, and presumably 20 times the total weight of extraction fluid was used. EPA Method 1311,



sections 7.2 and 7.2.14 describe filtering, extraction of the solids on the filter paper, and then recombining the filtrate and extract after tumbling. Alternatively, the filtrate and the extract could have been analyzed separately and the results combined mathematically.

Reply 4.3.2.3 Paragon has revised TCLP forms and SOPs to address the multiphasic requirements. Please see **Attachment G** for a copy of these TCLP forms and SOPs.

Item 4.3.3 Several findings were noted for the analysis of Total Petroleum Hydrocarbons:

Item 4.3.3.1 The Standard Operating Procedure for TPH-Gas and BTEX stated that screening is done before analysis in order to find an appropriate dilution. When asked, the analysts stated that no instrumental screening was being performed, but that he routinely screened samples using the 'sniff test' to establish dilutions. If true, the SOP should be revised to reflect actual practices and the analyst counseled about safety.

Reply 4.3.3.1 The TPH-Gas and BTEX SOP incorrectly stated that an initial screen is performed to determine appropriate dilution. All GC Fuels SOPs have been revised to reflect actual practice. Please see **Attachment I** for copies of the GC Fuels SOPs (SOP 406, TEPH/DRO; SOP 424, BTEX; SOP 425, TVPH/GRO).

On December 07, The QA Manager counseled the fuels/BTEX analyst against performing the "sniff test" on any sample and instructed the analyst to open all samples and standards in the portable fume hood located in the fuels laboratory.

Item 4.3.3.2 When analyzing methanol extracts by Purge and Trap, up to 1 mL of methanol might be added to a 5-mL sparger tubes. Typically, no more than 100 uL of the methanol is added to the sparger tube unless the calibration standards include similar amounts of methanol.

Reply 4.3.3.2 Paragon believes that there may have been a misunderstanding with regard to this item. Review of runlogs indicates that no more than 100 uL of methanol has been added to a sparger tube while performing a methanolic dilution of a sample. The QA Manager discussed this item with the Organics Manager, Chromatography Supervisor, and Fuels Analyst on December 10 and they were



aware that adding methanol in excess of 100 uL may strip an analytical column. These three (3) employees verified that they had not added more than 100 uL of methanol to a sparger tube while performing a methanolic dilution of a sample.

Item 4.3.3.3 TPH-G and TPH-D standards were stored in the freezer compartment of the refrigerator that contained samples to be analyzed for TPH-G and BTEX.

Reply 4.3.3.3 Paragon is aware that standards and samples must be stored separately to prevent cross contamination. On December 07, the QA Manager verified that this practice is in place throughout the laboratory. In particular, the TPH-G and TPH-D standards are stored in Unit #9 (freezer) and the environmental samples intended for fuels analysis are stored in Unit #10 (refrigerator).

Item 4.3.4 Laboratory practices were inconsistent with EPA Method 8310. The analyst stated that the lower value from the two detectors was the reported value for the analysis of Polynuclear Aromatic Hydrocarbons (PAHs) by HPLC. As illustrated in EPA Method 8310, Table 1, Naphthalene, Acenaphthylene, Acenaphthene, and Fluorene should be reported from the UV detector and all other PAHs from the Fluorescence detector.

Reply 4.3.4 On December 08, The QA Manager inspected various calibration files and discussed Method 8310 requirements with the two (2) HPLC analysts. From these reviews and conversations, it was determined that the four PAH compounds noted above are always quantitated from the UV detector (wavelengths examined at 254 nm and 280 nm). Acenaphthylene does not fluoresce and quantitative calibrations are not developed for the other three (3) compounds. Regarding the analysts' comment that the "lower value" was reported, the QA Manager has instructed them to follow the guidance given by SW-846, Section 7.10.4, "Comparison Between Results from Different Columns or Detectors." Please see *Attachment J* for documentation of the conversation.

Item 4.3.5 In observing the metals digestion process, the assessor noted that matrix spikes were added to soil after the addition of water to the sample.

Reply 4.3.5 Paragon understands that the intent of SW-846 is to add spiking compound to the matrix, not the reagent/solvent. On December 10, The QA Manager instructed the



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metals analyst, supervisor, and Technical Manager that that spiking solution shall be added to the soil sample before the addition of water to the sample. This operational practice has been changed as of December 10. Please see *Attachment J* for documentation of this conversation.

Item 4.3.6 Two findings were noted in the analysis of flash point:

Item 4.3.6.1 When analyzing soils for flash point, the assessor was told that cup of the flash point apparatus was packed with soil up to the line and the soil was not stirred. The Pensky-Martin flash point apparatus is approved for liquids with surface films and/or high-suspended solids, but there is no approved method for soils and solid materials.

Comment Some laboratories have developed modifications to the method using slurries that can be stirred, but these modifications should not be called a flash point by EPA Method 1010.

*Reply 4.3.6.1 Paragon concurs that SW-846 Method 1010 is written for a liquid matrix (Section 1.0, Method 1010). Paragon presents a modified approach in quoting flash point determination for solids that is based on an internal SOP. Please see *Attachment K* for a current list of capabilities that describes modified methods with a suffix (e.g., Method 1010M).*

Item 4.3.6.2 Samples were reported outside the acceptance criteria for the p-xylene. The p-xylene reference standard flash point determination is 27 ± 0.8 °C (81 ± 1.5 °F). On August 19, 1998, samples were reported when the reference check was 23 °C, and on July 20, 1998 when the reference check was 21 and 20.5 °C.

Reply 4.3.6.2 From experience and reading (1) SW-846, Method 1010 and (2) D 93-80, Test Methods For Flash Point by Pensky-Martens Closed Tester, ASTM publication, Paragon understands that flashpoint of the p-xylene standard depends on various factors (e.g., temperature, altitude/pressure). These factors explain the lower flash point for p-xylene reported by Paragon. In reviewing Paragon's logbook, it is seen that p-xylene routinely flashes at 20 - 24 °C as a result of lower pressure in Fort Collins and that daily corrections for barometric pressure are applied to the readings (cf. water boils at 100 °C at sea level and at 94-95 °C at higher elevations such as Fort Collins, CO). Paragon believes that our practice of flash point determination is compliant with the two (2)



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references cited above. Please see *Attachment L* for logbook pages that substantiate the temperature readings for p-xylene. Paragon notes that there is no calibration to be performed. Rather, the temperature is simply read from a thermometer.

Item 4.3.7 Violations of Good Laboratory Practices were not widespread, but occasional violations were noted. In responding to this finding, the laboratory should not respond to each example listed here as 4.3.7.1 through 4.3.7.5 but instead should describe its measurable approach to improved Good Laboratory Practices.

Item 4.3.7.1 Indelible ink was not always in logbooks.

Item 4.3.7.2 Some obliteration was observed.

Item 4.3.7.3 One analyst described recording data on scrap paper and later transcribing it into the analytical log, then discarding the original paper. In another case, an injection log was noted that it was a copy and the original was missing.

Item 4.3.7.4 Two vendor certifications for standards could not be located, and five expired standards were noted in the organics labs. When two analysts were asked if there was any expired standards stashed in the laboratory, they were clearly uncomfortable and hesitant in their answers, and then were relieved when the assessor said that he wouldn't ask where the standards were.

Item 4.3.7.5 Corrections did not always include initials and dates.

Reply 4.3.7 Paragon concurs that these Good Laboratory Practices must be reviewed with all employees. The QA Manager addressed these issues at the all-staff meeting on Tuesday, December 15. All employees were required to sign the policy memo that outlined requirements for using indelible ink, proper correction technique, preservation of original documentation, and handling/disposing of expired standards. These memos have been placed in each employee's training file. Please see *Attachment M* for a copy of the memo.

Item 4.3.8 Three findings were noted regarding temperature documentation:

Item 4.3.8.1 The Infrared (IR) thermometer utilized in Sample Control was "compared" to a calibrated thermometer in a cold storage unit



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but no temperature adjustment is made when there is a temperature difference between the two thermometers. It is also questionable if the temperature comparison is accurate since the IR thermometer temperature is taken from a "blank" spot in the cold storage unit and the other temperature is take from a thermometer immersed in liquid.

Reply 4.3.8.1 Paragon is not able to calibrate the portable IR temperature "gun," as it must be calibrated and certified by an independent, qualified vendor (c.f. balance calibration). The annual calibration and NIST certification was performed by Raytek on 11/23/98 (certificate #55697). Please note that Paragon's twice daily comparison is made to two (2) NIST certified thermometers immersed in liquid. Comparison to a "blank" spot in the refrigerator is not performed by Paragon. Please see *Attachment N* for copies of the Raytek NIST certificate and pages from the logbook.

Item 4.3.8.2 Temperature excursions were noted with cold storage units and corrective actions often noted; however, no closure was noted.

Reply 4.3.8.2 Paragon concurs that closure must be noted in the logbook.. The QA Manager has recently revised logbook forms to include instructions for notifying the QA Manager if temperature excursions occur so that Paragon may ensure complete documentation of temperature excursions. Please see *Attachment O* for copies of these forms.

Item 4.3.8.3 In the organics sections, thermometers were labeled with correction factors, but the dates of calibration were not listed.

Reply 4.3.8.3 Paragon acknowledges that thermometers throughout the laboratory require re-certification and labeling. The QA Department sent Paragon's reference thermometers to Ertco, in West Patterson, NJ for certification. As soon as the reference thermometers are returned, all laboratory thermometers will be standardized against the certified ones and dated tags affixed to each one. Please see *Attachment P* for documentation of certification by Ertco.



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Item 4.3.9 Regarding software quality assurance, there were four findings:

Item 4.3.9.1 The laboratory maintained a Disaster Recovery Plan for the Information Services Department. This plan addressed most of the quality assurance requirements for software issues that are outlined in the Navy Installation Restoration Laboratory Quality Assurance Guide; however, the manual did not address the policies and practices for the development, procurement, modification, maintenance, and use of computer software.

Item 4.3.9.2 The Disaster Recovery Plan does not address software validation and verification prior to use.

Item 4.3.9.3 No documentation was provided to demonstrate that the elements of the Plan (e.g., archiving) were in place.

*Reply 4.3.9.1 - 4.3.9.3 Paragon concurs that the information requested is not presented in the Disaster Recovery Plan given to QBD for review. To answer the question of software validation, Paragon notes that we do not participate in beta testing of software (e.g., HP Enviroquant, WARD, Alpha Vision). Paragon requires that all vendor software has been tested and approved by the manufacturer prior to purchase; therefore, we believe the risk of error is minimal. All vendor laboratory software is "locked" and Paragon does not have source codes to edit equations/algorithms. Please note that Paragon performs manual recalculation verifications **for each work order** to ensure that the instrument/software calculations are correct and can be created. Examples or recalculations from each department are presented in **Attachment Q**. In addition, Paragon presents two (2) SOPs and a recent policy memo (form attached) that address the issue of software validation. We believe that these documents address the requirements of Section 3.1.2.17 of the February 1996 manual.*

Item 4.3.9.4 Validation of software had either not been performed in the BTEX/Fuels Laboratory, or the equations and macros of the spreadsheet had not been secured against accidental or deliberate changes. On a summary page for the BTEX initial calibration by EPA Method 8021 for September 22, 1998, which used a spreadsheet macro, the Percent Relative Standard Deviations (%RSD) for Toluene was 28%, which was out of the acceptance



criteria. However, the actual %RSD used by the chromatography software was within criteria.

Reply 4.3.9.4 On December 09, the Chromatography Supervisor, Organics Manager, and Fuels Analyst reviewed the equations and macros used by the Fuels analyst for the September 22 analyses. They discerned that the problem noted above occurred because the Fuels analyst analyzed a 7-point curve but deleted the two highest level standards. The analyst did not import the data properly from the instrument to the spreadsheet, which caused the %RSD error. Paragon perceives this situation to be a training issue -- not a software validation issue -- and we believe that it has been addressed effectively.

In response to general software validation issues suggested by this item, please see the documentation provided in *Attachment Q*.

Item 4.3.10 The laboratory's Quality Assurance (QA) program has a foundation established, in that staff are cognizant of Quality Control requirements, the paper trail, expiration dates, maintenance, and similar activities. However, the QA program is incomplete:

Item 4.3.10.1 There was no written training program in use in the laboratory. Training files were not kept up-to-date. The laboratory's Quality Assurance Plan describes the responsibilities and types of documentation required, but these practices have not been adhered to. Department supervisors stated that their responsibilities and documentation requirements differed from that described in Section 14.2 of the Quality Assurance Plan. Standard operation procedures (SOPs) were available in the laboratory and staff could locate them; however, in some cases the staff did not know which book contained the specific SOP for the test that they were performing and, when the SOP was located, they were not familiar with the contents of the SOP.

Reply 4.3.10.1 Paragon acknowledges that the documentation of a training program and employee training files have not been maintained as a result of insufficient resources. The new QA Manager has begun training sessions (on a per method basis). To date, Method 8260B has been discussed at three separate sessions (11/06, 11/19, 11/20) and Method 8015M (DRO and GRO) training sessions



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have been completed (12/15 and 12/21). The next methods training sessions scheduled are: 8270C, 8081, 8082, 8151, 6010B, and 9056. Please see *Attachment R* for documentation of Method 8015M training.

The following documents will be added to each employee's training file: signed Code of Ethics Statement, personnel questionnaire, transcript or diploma, health and safety training, QA training, radiation safety training, SOP/Method training, IPR/DOC training, and off-site training. Examples of these documents follow for your review (*Attachment R*).

Distribution of SOPs has been revised by the QA Manager to enable every employee to find and review an SOP as needed. In addition to maintaining the three (3) master sets of controlled SOPs, the following changes have been made: (1) each group has been given a binder of SOPs needed for their use (e.g., GC/MS Volatiles analysts have been provided copies of all 500 series (GC/MS) SOPs and relevant 300 series (general chemistry) SOPs); (2) a Table of Contents that describes every SOP in the binder has been placed in the front of each binder; (3) the QA Manager sends e-mail to all employees, announcing the update/distribution of every SOP; and (4) the Table of Contents of all current SOPs has been distributed to each department.

Item 4.3.10.2 Internal audits have not been performed at the frequency described in the QA plan.

Reply 4.3.10.2 Paragon acknowledges that internal audits have not been performed routinely as a result of insufficient resources. However, Paragon notes that we receive several annual audits from state and federal agencies. Since October 1997, Paragon has been audited by the following 10 groups: US DOE Albuquerque / LANL SMO; US DOE Las Vegas / IT Corporation; State of California / Radiochemistry, State of California / conventional chemistry; State of Utah; State of Arizona; USACE / MRD; AFCEE / RUST E&I; US DOE Albuquerque / ESH-17; and NFESC / QBD. These agencies represent a diverse group of auditors and Paragon relies on their expertise to ensure continued production of compliant data.



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Paragon has recently hired an experienced QA analyst, Ms. Debra Scheib, to assist in performing QA functions. Her primary functions include: revising SOPs, performing internal audits, and reviewing 5% of Paragon's data. Following the update of all SOPs, Ms. Scheib will begin performing internal audits. Ms. Scheib's resume follows for your review (please see *Attachment S*). In addition, Section 11.1 of the LQAP has been rewritten to reflect actual practice (LQAP enclosed under separate cover).

Item 4.3.10.3 Routine quality assurance reports to management have not been performed.

Reply 4.3.10.3 The QA Manager has compiled a quality assurance report that includes the following items: state and federal certifications; performance evaluations results and responses (WS, WP, MAPEP, EML, EMSL for 1996 - 1998); external audits (10); List of SOPs; list of MDL studies; organizational chart; resumes; list of major instrumentation; and a list of capabilities. These documents will be provided upon request.

The quality assurance report will be updated every six (6) months. Please see Section 12 of the revised LQAP for additional information.

Item 4.3.10.4 Controlled documents were identified but there was no tracking system that identifies which person has received a particular document and any revisions or updates. In particular, there was no documentation that staff has received or read the Quality Assurance Manual or the SOPs.

Reply 4.3.10.4 Paragon acknowledges that the distribution of controlled documents has not been maintained. The previous QA Manager had developed and maintained a distribution list for the LQAP and this list will be revised upon distribution of the new LQAP. The current QA Manager has developed a distribution list that documents issuance of SOPs. Please see *Attachment T* for copies of these documents.

Item 4.3.10.5 Control charts were used only to generate upper and lower control limits and are not routinely in use in the laboratory. At



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least two sections were using surrogate limits that were dated before October 1997. The laboratory's Quality Assurance Plan includes a good description of runs, trends, and periodicity, which is not in practice in the laboratory.

Reply 4.3.10.5 Intralaboratory historical limits have been updated (February and March, 1999) and distributed to each analytical group and all Project Managers. *Attachment U* provides an example of updated qc limits (others are available upon request).

Please note that Paragon's federal and commercial clients frequently prescribe qc limits to be used for individual projects, so the laboratory's limits are not frequently used. Paragon's LIMS includes client and project specific qc limits for surrogates and spiking compounds; therefore, data are evaluated and reported against the correct qc limits as prescribed by our clients.

Item 4.3.10.6 *The laboratory did not have an effective corrective action system that was well defined and operational which 1) differentiates between major and minor QA exceedences and problems, and their appropriate corrective actions, 2) allows monitoring of the status of actions and documents their completion, and 3) tracks and identifies trends and recurrent issues. The laboratory did have a system using Non-conformance Reports (NCR) and a policy in the Quality Assurance Plan, but the practice and documentation was not consistent throughout the laboratory. As examples, 1) written requests for re-extractions were given to the Organics Preparation laboratory, but were not tracked for systematic trends, and there was no formal procedure for communication these exceedences to the client (when appropriate). 2) Surrogate failures were not tracked for trends that might indicate that staff need retraining or that the spiking standard is going bad.*

Reply 4.3.10.6 Paragon concurs that the practice of completing an NCR report and understanding of the document is inconsistent throughout the laboratory. The SOP that addresses the mechanics of completing NCRs, # 928, has been revised and distributed to all employees. The QA Manager reviewed the document with employees at an all-staff meeting on Tuesday, January 19, to ensure that all operations personnel understand how and when to complete an



NCR.. *Attachment V* includes a copy of the revised SOP, NCR form, and the memo from the January 19 meeting.

Item 4.3.10.7 Method detection limit (MDL) studies were in progress but some, such as TPH-Diesel (a.k.a. Diesel Range Organics) and EPA Method 8270 for soils, were out of date. A schedule for updating the MDL studies should be established and adhered to.

Reply 4.3.10.7 The QA Manager has developed a schedule for the completion of MDL studies. For tracking purposes, MDL studies have been logged in as work orders and appear on every group's work list. Copies of the QA Manager's MDL Schedule and completed, current MDL summaries follow for your review (please see Attachment W).

4.3.11 Quality Assurance Management Plan Review

Comment: Although this report describes findings regarding the QA Plan, it is acknowledged that SOPs and/or other policy documents might be available and current which provide the necessary information and which were not reviewed by the Assessors. Using a QA Plan as a general policy document and an SOP for the specific "how to" procedures is acceptable. Where appropriate to respond to critical, major, and minor deficiencies, the QA Plan may be revised to either 1) include the information that is currently is a SOP; 2) state that the topic is addressed in a certain section of each individual SOP (e.g., "QC acceptance criteria for each method is described in Section XX.YY of the analytical SOP for that method"); or 3) include a reference in the QA Plan that additional information in a particular document or SOP.

Information regarding the Navy requirements for Quality Assurance Plans are found in the Navy Installation Restoration Laboratory Quality Assurance Guide, Section 3.1.

Item 4.3.11.1 The QA Plan was last revised and approved on April 28, 1997. The QA Plan is in need of updating. The following are examples of items that are out-of-date:

The quality control requirements for several methods have been updated and any revisions should include updates to Sections 6 and 7 of the laboratory's QA Plan. For example, EPA Method



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6010B has loosened the acceptance criteria for the ICV from 5% to 10%.

Policy regarding real time assessment of control charts is not consistent with current lab practices (as noted in finding 4.3.1.5).

Reply 4.3.11.1 Quality control requirements have been reviewed and updated as appropriate. Please see the revised LQAP, which is enclosed under separate cover.

As stated in Reply 4.3.10.5, control limits have been updated and distributed. Example control charts are submitted as *Attachment U*. Accuracy and precision values shown in the LQAP are representative only. See Section 3.14 of the LQAP for a description of limits presented in the LQAP.

Item 4.3.11.2 The definition of a batch in Section 9.1 of the QA Plan is unacceptable because it allows some samples to be considered as a batch even when they may not be processed as a unit. For example, this definition allows samples that are extracted on two different days to be considered as a batch for quality control purposes (spiking and blanks) even if the first day's extraction is completed before the second day's samples are started.

Reply 4.3.11.2 Section 9.1 of the LQAP has been rewritten to correct the definition of batch.

Item 4.3.11.3 The definition of the Method Detection Limit in Section TOC (Terms and Abbreviations) is incomplete. The definition describes how it is calculated but not what the detection limit actually is, i.e., the smallest amount that may be detected at a given statistical confidence level.

Reply 4.3.11.3 The definition of method detection limit has been clarified in Section TOC of the LQAP.

Item 4.3.11.4 Control limits for TPH-Diesel as listed in Table 3-1 are greater than that allowed by some states for which the laboratory might be performing analysis of samples for the Navy. For example, the limits listed in the table are 30-150% for waters, but several states on the West Coast mandate acceptance criteria of 50-150%.



Reply 4.3.11.4 As stated above, control limits have been updated and distributed. Please note that Paragon's federal and commercial clients frequently prescribe qc limits to be used for individual projects, so the intralaboratory historical limits are not frequently used. Paragon's LIMS includes client and project specific qc limits for surrogates and spiking compounds; therefore, data are evaluated and reported against the correct qc limits.

Item 4.3.11.5 Tables 3-5 through 3-12 contain either no acceptance limits for some compounds or guidance/advisory limits listed in several methods. Since most of these methods require periodic updating of historical acceptance criteria, and since the Navy Installation Restoration Laboratory Quality Assurance Guide requires that all analytes of concern be spiked, these tables should be updated to reflect the acceptance criteria in practice.

Reply 4.3.11.5 Please see the control charts included in Attachment U for updated values. Paragon routinely spikes the full list of compounds and evaluates blank spikes and matrix spikes for all compounds.

Item 4.3.11.6 Section 6.4.2 and 9.8.5 of the QA Plan regarding second column confirmation of gas chromatographic analyses are not consistent with guidance in EPA Method 8000b, Sections 7.9 and 7.10.4.

Reply 4.3.11.6 These sections have been rewritten per SW-846 guidance.

Item 4.3.11.7 The assessors could not locate several items in the QA Plan: Procedures used in the event of temporary absence of key personnel.

Document archival is addressed for raw data and reports but not for QA documentation. Retention of a history of SOP revisions, expired SOPs, expired QA Plans, training records, etc. is not addressed.

the frequency and procedures for the review of controlled documents.

Identification of the person responsible for the documentation of Data Quality Objectives.



The frequency of blind performance evaluation samples and internal performance studies.

Calibration and preventive maintenance for thermometers and pipettors. A SOP was available for the standardization of thermometers.

The QA Plan has a procedure for the oversight of subcontracting laboratories but does not list the criteria for hiring a subcontracting laboratory or the criteria for the acceptance of their data. It should also be noted that Navy samples may not be subcontracted to laboratory that has not successfully completed the NFESC evaluation process.

The frequency of internal audits.

Security for confidential information and audit trails for data changes.

Reply 4.3.11.7 Paragon sincerely appreciates the auditor's detailed list of needed corrections / updates to the laboratory's LQAP. These comments and resultant changes have been incorporated into Revision 4 of Paragon's LQAP.

Item 4.3.12 *On the master list of Standard Operating Procedures, 14 of 277 SOPs had not been reviewed and/or updated within the previous 12 months. It is assumed that many of these un-reviewed SOPs are no longer in use. The SOPs that are not in use should be noted as inactive or canceled, and a schedule established for reviewing, editing, and approving the remaining SOPs.*

Reply 4.3.12 Paragon acknowledges that many SOPs require revision. Several SOPs have been retired. Paragon has provided a revision schedule and updated Table of Contents to QBD. As of this writing, 64 SOPs have been revised. Upon request, we will provide copies of any updated SOP.

Paragon understands that Ms. Patti Moreno, of NFESC, will continue to monitor the progress of SOP updates following closure of this audit by QBD.

Item 4.3.13 *As part of the audit process a subset of the laboratory's SOPs were reviewed for technical soundness and adherence to Navy QA Program requirements. Copies*



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of the SOPs listed in the following table were provided to QBD and were reviewed after the on-site audit.

Item 4.3.13.1 The SOP review and approval process was not consistently followed. SOP No. 804Rev4, Digestion of Waters, Soils and Wastes for Metals Analysis, had been updated by the metals preparation technician to reflect the most recent promulgated method. The notes and changes made on the SOP were in effect but had not been reviewed or approved by the appropriate staff members.

Reply 4.3.13.1 Paragon acknowledges that significant changes (such as SW-846 updates) require a revision of the SOP and that handwritten, unofficial notes are not sufficient. The QA Manager has scheduled annual updates of all SOPs, which -- in conjunction with training sessions -- we believe will ensure compliant SOP review and approval process throughout the laboratory.

Item 4.3.13.2 The following general comments refer to all SOPs reviewed by QBD's offices or in the laboratory:

The SOPs that were prepared prior to 1997 that were reviewed by the auditors were technically weak and contained inadequate instructions for instrument of computer set-up, operation, and shut down. These include the SOPs for BTEX and TPH-G (#426) and Extractable Hydrocarbons(#406).

Comment: A SOP should be complete enough for use as a training tool for new analysts, for a refresher or guide for an

experienced analyst, and, when combined with analysis logs, will allow complete recreation of the test by third parties. SOPs that were recently reviewed and updates, such as Gasoline (#425), Semivolatiles (#506), and Volatile Organics (#525) were far superior, complete, and comprehensive.

Several of the SOPs failed to list the calculations for water and soil sample concentrations. The calculations should be given to check the performance of the software and to allow recreation of the test at a future date. This finding includes the SOPs for Chlorinated Pesticides (#402) and PAHs (#400).



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Several of the SOPs failed to describe the preparation of standards, either not describing the flasks and pipettes, and/or failing to describe the initial and final volumes and concentrations to be used.

Item 4.3.13.3 RE: Polynuclear Aromatic Hydrocarbons -- Method 8310/610 In addition to the findings described in 4.3.13.1, this SOP established fixed retention time windows. The width of a retention time window for HPLC should be established as described in Section 9.8.6 of the laboratory's Quality Assurance Plan.

Reply 4.3.13 Paragon concurs with the auditor's comments regarding SOPs. Each item will be addressed as is appropriate during SOP revisions. Copies of revised SOPs are available upon request.

Paragon extends its thanks and appreciation to Quality By Design for the thorough and detailed audit report and the time that the auditors spent discussing requirements with our staff. The auditor's findings have enabled Paragon to make necessary changes to its systems and processes, thereby ensuring that data quality objectives are met for our clients.

We hope that our responses meet your requirements. Please contact me at 970 490 1511 if you have any questions or need additional information.

Respectfully Submitted,

Debra Henderer
Quality Assurance Manager
Paragon Analytics, Inc.

Enclosures

ENCLOSURE 3:

Paragon MDLs

Paragon Analytics, Inc.

MDL for Method SW6010

Matrix: SOLID
Extraction Method: SW3050
AnalysisDate: 01/12/2001
ActiveDate: 01/26/2001
ExpireDate: 01/12/2002
Instrument: ICP
Units: UG/KG

MDL Comments:

RADIAL ICP

Analyte:	MDL:	Comments:
ALUMINUM	1630	
ANTIMONY	1750	
ARSENIC	3260	
BARIUM	61.5	
BERYLLIUM	27.8	
BORON	547	
CADMIUM	335	
CALCIUM	4290	
CHROMIUM	654	
COBALT	252	
COPPER	395	
IRON	757	
LEAD	4530	
LITHIUM	101	
MAGNESIUM	10100	
MANGANESE	62.1	
MOLYBDENUM	303	
NICKEL	1040	
PHOSPHORUS	8850	
POTASSIUM	4930	
SELENIUM	5000	
SILICON	1470	
SILVER	265	
SODIUM	2030	
STRONTIUM	26.6	
THALLIUM	7680	
TIN	2220	
TITANIUM	65.1	
VANADIUM	179	
ZINC	222	

Paragon Analytics, Inc.

MDL for Method SW6010

Matrix: SOLID
Extraction Method: SW3050
AnalysisDate: 01/23/2001
ActiveDate: 01/31/2001
ExpireDate: 01/23/2002
Instrument: ICPTrace
Units: UG/KG

MDL Comments:

AXIAL

Analyte:	MDL:	Comments:
ALUMINUM	815	
ANTIMONY	150	
ARSENIC	236	
BARIUM	12.6	
BERYLLIUM	12.3	
CADMIUM	19.3	
CALCIUM	817	
CHROMIUM	35.2	
COBALT	29.4	
COPPER	44.8	
IRON	922	
LEAD	155	
MAGNESIUM	507	
MANGANESE	22.7	
NICKEL	64.4	
POTASSIUM	2999	
SELENIUM	112	
SILVER	29.2	
SODIUM	1789	
THALLIUM	258	
URANIUM	9700	
VANADIUM	46.9	
ZINC	59.6	

Paragon Analytics, Inc.

MDL for Method SW6010

Matrix: LIQUID
Extraction Method: SW3005
AnalysisDate: 01/16/2001
ActiveDate: 01/26/2001
ExpireDate: 01/16/2002
Instrument: ICP
Units: UG/L

MDL Comments:
RADIAL ICP

Analyte:	MDL:	Comments:
ALUMINUM	16.4	
ANTIMONY	15.8	
ARSENIC	30.4	
BARIUM	0.669	
BERYLLIUM	0.323	
BORON	5.42	
CADMIUM	1.7	
CALCIUM	30.5	
CHROMIUM	3.46	
COBALT	1.54	
COPPER	2.24	
IRON	6.6	
LEAD	23.2	
LITHIUM	1.02	
MAGNESIUM	40.5	
MANGANESE	1.2	
MOLYBDENUM	3.95	
NICKEL	6.53	
PHOSPHORUS	56.5	
POTASSIUM	61	
SELENIUM	42	
SILICON	21.8	
SILVER	2.78	
SODIUM	26.5	
STRONTIUM	0.17	
THALLIUM	58.1	
TIN	13.7	
TITANIUM	1.04	
VANADIUM	2.23	
ZINC	5.4	

Paragon Analytics, Inc.

MDL for Method SW6010

Matrix: LIQUID
Extraction Method: SW3005
AnalysisDate: 12/08/2000
ActiveDate: 01/10/2001
ExpireDate: 12/08/2001
Instrument: ICPTTrace
Units: UG/L

MDL Comments:

TRACE / AXIAL

Analyte:	MDL:	Comments:
ALUMINUM	16.2	
ANTIMONY	1.59	
ARSENIC	2.4	
BARIUM	0.306	
BERYLLIUM	0.116	
CADMIUM	0.135	
CALCIUM	4.04	
CHROMIUM	0.306	
COBALT	0.169	
COPPER	0.718	
IRON	7.59	
LEAD	1.16	
MAGNESIUM	4.67	
MANGANESE	0.114	
NICKEL	0.656	
POTASSIUM	69.1	
SELENIUM	2.32	
SILVER	0.348	
SODIUM	53.7	
THALLIUM	2.83	
URANIUM	46	
VANADIUM	0.474	
ZINC	0.854	

Paragon Analytics, Inc.

MDL for Method SW6010

Matrix: LIQUID
Extraction Method: SW3010
AnalysisDate: 12/14/2000
ActiveDate: 01/26/2001
ExpireDate: 12/14/2001
Instrument: ICP
Units: UG/L

MDL Comments:
RADIAL ICP

Analyte:	MDL:	Comments:
ALUMINUM	226	
ANTIMONY	195	
ARSENIC	340	
BARIUM	8.48	
BERYLLIUM	7.85	
CADMIUM	18.3	
CALCIUM	302	
CHROMIUM	42.3	
COBALT	21.6	
COPPER	26.2	
IRON	123	
LEAD	327	
MAGNESIUM	806	
MANGANESE	20.9	
NICKEL	102	
POTASSIUM	582	
SELENIUM	498	
SILVER	16	
SODIUM	53.7	
THALLIUM	816	
URANIUM	950	
VANADIUM	25.3	
ZINC	57.5	

Paragon Analytics, Inc.

MDL for Method SW6010

Matrix: LIQUID
Extraction Method: SW3010
AnalysisDate: 12/08/2000
ActiveDate: 01/10/2001
ExpireDate: 12/08/2001
Instrument: ICPTTrace
Units: UG/L

MDL Comments:
TRACE / AXIAL

Analyte:	MDL:	Comments:
ALUMINUM	323	
ANTIMONY	27.7	
ARSENIC	18.4	
BARIUM	1.23	
BERYLLIUM	4.77	
CADMIUM	1.35	
CALCIUM	59.4	
CHROMIUM	4.43	
COBALT	7.85	
COPPER	4.82	
IRON	113	
LEAD	11	
MAGNESIUM	28.5	
MANGANESE	0.785	
NICKEL	5.71	
POTASSIUM	2700	
SELENIUM	17.8	
SILVER	7.3	
SODIUM	53.7	
THALLIUM	22.9	
URANIUM	950	
VANADIUM	5.18	
ZINC	31.2	

Paragon Analytics, Inc.

MDL for Method SW7196

Matrix: SOLID
Extraction Method: NONE
AnalysisDate: 01/26/2001
ActiveDate: 02/14/2001
ExpireDate: 01/26/2002
Instrument: Spec
Units: UG/KG

MDL Comments:

Analyte:	MDL:	Comments:
CHROMIUM VI	39.3	

Paragon Analytics, Inc.

MDL for Method SW7196

Matrix: LIQUID
Extraction Method: NONE
AnalysisDate: 01/26/2001
ActiveDate: 02/14/2001
ExpireDate: 01/26/2002
Instrument: Spec
Units: UG/L

MDL Comments:

Analyte:	MDL:	Comments:
CHROMIUM VI	1.45	•

Paragon Analytics, Inc.

MDL for Method SW7196

Matrix: SOLID
Extraction Method: SW3060
AnalysisDate: 02/20/2001
ActiveDate: 03/05/2001
ExpireDate: 02/20/2002
Instrument: Spec
Units: UG/KG

MDL Comments:

Analyte:	MDL:	Comments:
CHROMIUM VI	635	

Paragon Analytics, Inc.

MDL for Method SW7471

Matrix: SOLID
Extraction Method: METHOD
AnalysisDate: 01/19/2001
ActiveDate: 01/26/2001
ExpireDate: 01/19/2002
Instrument: LEEMAN
Units: UG/KG

MDL Comments:

Analyte:	MDL:	Comments:
MERCURY	1.95	

Paragon Analytics, Inc.

MDL for Method SW7470

Matrix: LIQUID
Extraction Method: METHOD
AnalysisDate: 12/21/2000
ActiveDate: 01/11/2001
ExpireDate: 12/21/2001
Instrument: LEEMAN
Units: UG/L

MDL Comments:

Analyte:	MDL:	Comments:
MERCURY	0.0118	

Paragon Analytics, Inc.

MDL for Method SW8015MCALUFT

Matrix: SOLID
Extraction Method: METHOD
AnalysisDate: 03/07/2001
ActiveDate: 03/07/2001
ExpireDate: 03/07/2002
Instrument: FUELS-1
Units: UG/KG

MDL Comments:

CAL LUFT MDL for DRO. Prep per CAL LUFT method. 20 g = initial wt. 5 mL = FV. Shake, 4 hr. Value for JP-5 not updated. DBH 03/13/01.

Analyte:	MDL:	Comments:
DIESEL RANGE ORGANICS	1530	
JP-5	10.3	
MOTOR OIL RANGE ORGANICS	1120	
TOTAL EXTRACTABLE PETROLEUM HYDROCARBO	1530	

Paragon Analytics, Inc.

MDL for Method SW8015M

Matrix: LIQUID
Extraction Method: METHOD
AnalysisDate: 01/20/2001
ActiveDate: 01/31/2001
ExpireDate: 01/31/2002
Instrument: FUELS-1
Units: UG/L

MDL Comments:

Extraction per SOP 603, modified. 80 mL to 4 mL. RL = 1.0 ppm.

Analyte:	MDL:	Comments:
DIESEL RANGE ORGANICS	206	
TOTAL EXTRACTABLE PETROLEUM HYDROCARBO	206	

Paragon Analytics, Inc.

MDL for Method SW8015MLOW

Matrix: LIQUID

Extraction Method: METHOD

AnalysisDate: 01/20/2001

ActiveDate: 01/26/2001

ExpireDate: 01/20/2002

Instrument: FUELS-1

Units: UG/L

MDL Comments:

Extraction per SOP 603. 160 mL to 4 mL. RL = 0.5 ppm. IT LV uses this extraction/MDL.

Analyte:	MDL:	Comments:
DIESEL RANGE ORGANICS	35.6	
TOTAL EXTRACTABLE PETROLEUM HYDROCARBO	35.6	

Paragon Analytics, Inc.

MDL for Method SW8081

Matrix: SOLID
Extraction Method: SW3540
AnalysisDate: 02/23/2001
ActiveDate: 04/10/2001
ExpireDate: 02/23/2002
Instrument: Pest-1
Units: UG/KG

MDL Comments:

toxaphene only.

Analyte:	MDL:	Comments:
TOXAPHENE	9.77	

Paragon Analytics, Inc.

MDL for Method SW8081

Matrix: LIQUID
Extraction Method: SW3520
AnalysisDate: 02/23/2001
ActiveDate: 04/10/2001
ExpireDate: 02/23/2002
Instrument: Pest-1
Units: UG/L

MDL Comments:

technical chlordane and toxaphene only

Analyte:	MDL:	Comments:
CHLORDANE	0.0513	
TOXAPHENE	0.492	

Paragon Analytics, Inc.

MDL for Method SW8082

Matrix: SOLID
Extraction Method: SW3540
AnalysisDate: 02/09/2001
ActiveDate: 04/02/2001
ExpireDate: 02/09/2002
Instrument: PEST-1
Units: UG/KG

MDL Comments:

Sulfuric acid cleanup performed for MDL study, as for all samples.

Analyte:	MDL:	Comments:
AROCLOR-1016	5.74	
AROCLOR-1221	9.7	
AROCLOR-1232	5.27	
AROCLOR-1242	5.19	
AROCLOR-1248	5.68	
AROCLOR-1254	3.29	
AROCLOR-1260	7.49	

Paragon Analytics, Inc.

MDL for Method SW8082

Matrix: LIQUID
Extraction Method: SW3520
AnalysisDate: 02/09/2001
ActiveDate: 04/02/2001
ExpireDate: 02/09/2002
Instrument: PEST-1
Units: UG/L

MDL Comments:

Sulfuric acid cleanup performed for MDL study, as for all samples.

Analyte:	MDL:	Comments:
AROCLOR-1016	0.142	
AROCLOR-1221	0.259	
AROCLOR-1232	0.101	
AROCLOR-1242	0.124	
AROCLOR-1248	0.149	
AROCLOR-1254	0.0772	
AROCLOR-1260	0.0395	

Paragon Analytics, Inc.

MDL for Method SW8151

Matrix: LIQUID
Extraction Method: METHOD
AnalysisDate: 02/19/2001
ActiveDate: 05/15/2001
ExpireDate: 02/19/2002
Instrument: Herb-1
Units: UG/L

MDL Comments:

Analyte:	MDL:	Comments:
2,4,5-T	0.0147	
2,4-D	0.112	
2,4-DB	0.152	
DALAPON	0.408	
DICAMBA	0.0109	
DICHLOROPROP	0.324	
DINOSEB	0.0773	
MCPA	10.4	
MCPP	33	
SILVEX	0.0085	

Paragon Analytics, Inc.

MDL for Method SW8260

Matrix: SOLID
Extraction Method: SW5030
AnalysisDate: 03/14/2001
ActiveDate: 05/03/2001
ExpireDate: 03/14/2002
Instrument: HPV1
Units: UG/KG

MDL Comments:

5 g solid.

Analyte:	MDL:	Comments:
1,1,1,2-TETRACHLOROETHANE	1.18	
1,1,1-TRICHLOROETHANE	0.96	
1,1,2,2-TETRACHLOROETHANE	2.91	
1,1,2-TRICHLOROETHANE	1.32	
1,1-DICHLOROETHANE	0.95	
1,1-DICHLOROETHENE	0.91	
1,1-DICHLOROPROPENE	1.05	
1,2,3-TRICHLOROBENZENE	1.9	
1,2,3-TRICHLOROPROPANE	3.04	
1,2,4-TRICHLOROBENZENE	1.21	
1,2,4-TRIMETHYLBENZENE	1.23	
1,2-DIBROMO-3-CHLOROPROPANE	4.03	
1,2-DIBROMOETHANE	1.41	
1,2-DICHLOROBENZENE	1.16	
1,2-DICHLOROETHANE	1.53	
1,2-DICHLOROPROPANE	0.74	
1,3,5-TRIMETHYLBENZENE	1.51	
1,3-DICHLOROBENZENE	1.04	
1,3-DICHLOROPROPANE	1.24	
1,4-DICHLOROBENZENE	1.14	
1-CHLOROHEXANE	1.5	
2,2-DICHLOROPROPANE	1.13	
2-BUTANONE	18.6	
2-CHLOROETHYL VINYL ETHER	2.09	
2-CHLOROTOLUENE	1.52	
2-HEXANONE	17.1	
4-CHLOROTOLUENE	1.19	
4-METHYL-2-PENTANONE	16.3	
ACETONE	18.4	
ACROLEIN	28.7	
ACRYLONITRILE	31.6	
BENZENE	0.98	
BROMOBENZENE	1.58	
BROMOCHLOROMETHANE	0.95	

Paragon Analytics, Inc.

MDL for Method SW8260

Matrix: SOLID
Extraction Method: SW5030
AnalysisDate: 03/14/2001
ActiveDate: 05/03/2001
ExpireDate: 03/14/2002
Instrument: HPV1
Units: UG/KG

MDL Comments:

5 g solid.

Analyte:	MDL:	Comments:
BROMODICHLOROMETHANE	0.65	
BROMOFORM	1.62	
BROMOMETHANE	1.32	
CARBON DISULFIDE	0.92	
CARBON TETRACHLORIDE	0.99	
CHLOROBENZENE	1.04	
CHLOROETHANE	1.29	
CHLOROFORM	0.8	
CHLOROMETHANE	0.88	
CIS-1,2-DICHLOROETHENE	1.06	
CIS-1,3-DICHLOROPROPENE	1.23	
DIBROMOCHLOROMETHANE	1.1	
DIBROMOMETHANE	1.28	
DICHLORODIFLUOROMETHANE	1.15	
ETHYLBENZENE	1.02	
HEXACHLOROBUTADIENE	1.36	
IODOMETHANE	1.07	
ISOPROPYLBENZENE	1.29	
M+P-XYLENE	2.2	
METHYL TERTIARY BUTYL ETHER	1.92	
METHYLENE CHLORIDE	1.19	
N-BUTYLBENZENE	1.53	
N-PROPYLBENZENE	1.29	
NAPHTHALENE	2.41	
O-XYLENE	1.21	
P-ISOPROPYLTOLUENE	1.32	
SEC-BUTYLBENZENE	1.34	
STYRENE	1.18	
TERT-BUTYLBENZENE	1.29	
TETRACHLOROETHENE	1.2	
TOLUENE	0.94	
TRANS-1,2-DICHLOROETHENE	1.05	
TRANS-1,3-DICHLOROPROPENE	1.11	
TRICHLOROETHENE	0.94	

Paragon Analytics, Inc.

MDL for Method SW8260

Matrix: SOLID
Extraction Method: SW5030
AnalysisDate: 03/14/2001
ActiveDate: 05/03/2001
ExpireDate: 03/14/2002
Instrument: HPV1
Units: UG/KG

MDL Comments:

5 g solid.

Analyte:	MDL:	Comments:
TRICHLOROFLUOROMETHANE	1.71	
TRICHLOROTRIFLUOROETHANE	1.69	
VINYL ACETATE	2.96	
VINYL CHLORIDE	0.93	

Paragon Analytics, Inc.

MDL for Method SW8260 25

Matrix: LIQUID
Extraction Method: SW5030
AnalysisDate: 02/15/2001
ActiveDate: 05/15/2001
ExpireDate: 02/15/2002
Instrument: HPV1
Units: UG/L

MDL Comments:

25 mL purge.

Analyte:	MDL:	Comments:
1,1,1,2-TETRACHLOROETHANE	0.146	
1,1,1-TRICHLOROETHANE	0.131	
1,1,2,2-TETRACHLOROETHANE	0.197	
1,1,2-TRICHLOROETHANE	0.172	
1,1-DICHLOROETHANE	0.126	
1,1-DICHLOROETHENE	0.168	
1,1-DICHLOROPROPENE	0.112	
1,2,3-TRICHLOROBENZENE	0.332	
1,2,3-TRICHLOROPROPANE	0.346	
1,2,4-TRICHLOROBENZENE	0.398	
1,2,4-TRIMETHYLBENZENE	0.333	
1,2-DIBROMO-3-CHLOROPROPANE	0.872	
1,2-DIBROMOETHANE	0.24	
1,2-DICHLOROBENZENE	0.319	
1,2-DICHLOROETHANE	0.145	
1,2-DICHLOROPROPANE	0.131	
1,3,5-TRIMETHYLBENZENE	0.324	
1,3-DICHLOROBENZENE	0.334	
1,3-DICHLOROPROPANE	0.183	
1,4-DICHLOROBENZENE	0.304	
1-CHLOROHEXANE	0.24	
2,2-DICHLOROPROPANE	0.172	
2-BUTANONE	1.07	
2-CHLOROETHYL VINYL ETHER	0.289	
2-CHLOROTOLUENE	0.322	
2-HEXANONE	1.09	
4-CHLOROTOLUENE	0.312	
4-METHYL-2-PENTANONE	0.689	
ACETONE	3.13	
ACROLEIN	2.13	
ACRYLONITRILE	1.03	
BENZENE	0.12	
BROMOBENZENE	0.345	
BROMOCHLOROMETHANE	0.268	

Paragon Analytics, Inc.

MDL for Method SW8260 25

Matrix: LIQUID
Extraction Method: SW5030
AnalysisDate: 02/15/2001
ActiveDate: 05/15/2001
ExpireDate: 02/15/2002
Instrument: HPV1
Units: UG/L

MDL Comments:

25 mL purge.

Analyte:	MDL:	Comments:
BROMODICHLOROMETHANE	0.175	
BROMOFORM	0.264	
BROMOMETHANE	0.138	
CARBON DISULFIDE	0.196	
CARBON TETRACHLORIDE	0.175	
CHLOROBENZENE	0.171	
CHLOROETHANE	0.148	
CHLOROFORM	0.136	
CHLOROMETHANE	0.0927	
CIS-1,2-DICHLOROETHENE	0.112	
CIS-1,3-DICHLOROPROPENE	0.137	
DIBROMOCHLOROMETHANE	0.214	
DIBROMOMETHANE	0.181	
DICHLORODIFLUOROMETHANE	0.0797	
ETHYLBENZENE	0.224	
HEXACHLOROBUTADIENE	0.384	
IODOMETHANE	0.142	
ISOPROPYLBENZENE	0.338	
M+P-XYLENE	0.436	
METHYL TERTIARY BUTYL ETHER	0.194	
METHYLENE CHLORIDE	0.339	
N-BUTYLBENZENE	0.354	
N-PROPYLBENZENE	0.338	
NAPHTHALENE	0.421	
O-XYLENE	0.222	
P-ISOPROPYLTOLUENE	0.32	
SEC-BUTYLBENZENE	0.329	
STYRENE	0.218	
TERT-BUTYLBENZENE	0.333	
TETRACHLOROETHENE	0.233	
TOLUENE	0.153	
TRANS-1,2-DICHLOROETHENE	0.139	
TRANS-1,3-DICHLOROPROPENE	0.2	
TRICHLOROETHENE	0.17	

Paragon Analytics, Inc.

MDL for Method SW8260 25

Matrix: LIQUID
Extraction Method: SW5030
AnalysisDate: 02/15/2001
ActiveDate: 05/15/2001
ExpireDate: 02/15/2002
Instrument: HPV1
Units: UG/L

MDL Comments:
25 mL purge.

Analyte:	MDL:	Comments:
TRICHLOROFLUOROMETHANE	0.113	
TRICHLOROTRIFLUOROETHANE	0.153	
VINYL ACETATE	0.403	
VINYL CHLORIDE	0.0967	

Paragon Analytics, Inc.

MDL for Method SW8260

Matrix: LIQUID
Extraction Method: SW5030
AnalysisDate: 02/23/2001
ActiveDate: 05/15/2001
ExpireDate: 02/23/2002
Instrument: HPV1
Units: UG/L

MDL Comments:

5 mL purge.

Analyte:	MDL:	Comments:
1,1,1,2-TETRACHLOROETHANE	0.576	
1,1,1-TRICHLOROETHANE	0.741	
1,1,2,2-TETRACHLOROETHANE	0.924	
1,1,2-TRICHLOROETHANE	0.998	
1,1-DICHLOROETHANE	0.45	
1,1-DICHLOROETHENE	0.916	
1,1-DICHLOROPROPENE	0.562	
1,2,3-TRICHLOROBENZENE	1.04	
1,2,3-TRICHLOROPROPANE	1.15	
1,2,4-TRICHLOROBENZENE	0.863	
1,2,4-TRIMETHYLBENZENE	0.869	
1,2-DIBROMO-3-CHLOROPROPANE	1.32	
1,2-DIBROMOETHANE	1	
1,2-DICHLOROBENZENE	0.873	
1,2-DICHLOROETHANE	0.474	
1,2-DICHLOROPROPANE	0.687	
1,3,5-TRIMETHYLBENZENE	0.924	
1,3-DICHLOROBENZENE	0.894	
1,3-DICHLOROPROPANE	0.734	
1,4-DICHLOROBENZENE	0.805	
1-CHLOROHEXANE	0.898	
2,2-DICHLOROPROPANE	0.784	
2-BUTANONE	5.24	
2-CHLOROETHYL VINYL ETHER	3.55	
2-CHLOROTOLUENE	0.767	
2-HEXANONE	4.89	
4-CHLOROTOLUENE	0.814	
4-METHYL-2-PENTANONE	4.25	
ACETONE	7.69	
ACROLEIN	11	
ACRYLONITRILE	9.98	
BENZENE	0.441	
BROMOBENZENE	0.948	
BROMOCHLOROMETHANE	0.695	

Paragon Analytics, Inc.

MDL for Method SW8260

Matrix: LIQUID
Extraction Method: SW5030
AnalysisDate: 02/23/2001
ActiveDate: 05/15/2001
ExpireDate: 02/23/2002
Instrument: HPV1
Units: UG/L

MDL Comments:

5 mL purge.

Analyte:	MDL:	Comments:
BROMODICHLOROMETHANE	0.651	
BROMOFORM	0.453	
BROMOMETHANE	1.72	
CARBON DISULFIDE	0.849	
CARBON TETRACHLORIDE	0.705	
CHLOROBENZENE	0.638	
CHLOROETHANE	1.18	
CHLOROFORM	0.677	
CHLOROMETHANE	0.54	
CIS-1,2-DICHLOROETHENE	0.538	
CIS-1,3-DICHLOROPROPENE	0.66	
DIBROMOCHLOROMETHANE	0.819	
DIBROMOMETHANE	0.75	
DICHLORODIFLUOROMETHANE	0.811	
ETHYLBENZENE	0.567	
HEXACHLOROBUTADIENE	0.885	
IODOMETHANE	2.83	
ISOPROPYLBENZENE	0.844	
M+P-XYLENE	1.43	
METHYL TERTIARY BUTYL ETHER	0.8	
METHYLENE CHLORIDE	0.6	
N-BUTYLBENZENE	0.877	
N-PROPYLBENZENE	0.839	
NAPHTHALENE	0.861	
O-XYLENE	0.668	
P-ISOPROPYLTOLUENE	0.847	
SEC-BUTYLBENZENE	0.751	
STYRENE	0.665	
TERT-BUTYLBENZENE	0.852	
TETRACHLOROETHENE	0.798	
TOLUENE	0.467	
TOTAL XYLENES	0.668	MDL set to lowest isomer value for EDD reporting purposes
TRANS-1,2-DICHLOROETHENE	0.776	
TRANS-1,3-DICHLOROPROPENE	0.451	

Paragon Analytics, Inc.

MDL for Method SW8260

Matrix: LIQUID
Extraction Method: SW5030
AnalysisDate: 02/23/2001
ActiveDate: 05/15/2001
ExpireDate: 02/23/2002
Instrument: HPV1
Units: UG/L

MDL Comments:

5 mL purge.

Analyte:	MDL:	Comments:
TRICHLOROETHENE	0.796	
TRICHLOROFLUOROMETHANE	1.16	
TRICHLOROTRIFLUOROETHANE	1.17	
VINYL ACETATE	1.2	
VINYL CHLORIDE	0.856	

Paragon Analytics, Inc.

MDL for Method SW8270

Matrix: LIQUID
Extraction Method: SW3510
AnalysisDate: 02/05/2001
ActiveDate: 05/16/2001
ExpireDate: 02/05/2002
Instrument: HPSV1
Units: UG/L

MDL Comments:
separatory funnel / SW3510.

Analyte:	MDL:	Comments:
1,2,4-TRICHLOROBENZENE	2	
1,2-DICHLOROBENZENE	1.92	
1,3-DICHLOROBENZENE	1.9	
1,4-DICHLOROBENZENE	1.9	
2,4,5-TRICHLOROPHENOL	2.27	
2,4,6-TRICHLOROPHENOL	2.05	
2,4-DICHLOROPHENOL	2.21	
2,4-DIMETHYLPHENOL	1.55	
2,4-DINITROPHENOL	13.4	
2,4-DINITROTOLUENE	2.74	
2,6-DINITROTOLUENE	2.11	
2-CHLORONAPHTHALENE	2.82	
2-CHLOROPHENOL	1.99	
2-METHYLNAPHTHALENE	2.46	
2-METHYLPHENOL	1.8	
2-NITROANILINE	4.27	
2-NITROPHENOL	2.21	
3+4-METHYLPHENOL	1.4	calibration standard contains 4 meph only
3,3'-DICHLOROBENZIDINE	5.13	
3-NITROANILINE	10.6	
4,6-DINITRO-2-METHYLPHENOL	11.9	
4-BROMOPHENYL PHENYL ETHER	2.13	
4-CHLORO-3-METHYLPHENOL	2.48	
4-CHLOROANILINE	2.8	
4-CHLOROPHENYL PHENYL ETHER	2.71	
4-METHYLPHENOL	1.64	
4-NITROANILINE	15.4	
4-NITROPHENOL	6.75	
ACENAPHTHENE	2.52	
ACENAPHTHYLENE	2.1	
ANILINE	2.05	
ANTHRACENE	2.29	
AZOBENZENE	2.41	
BENZIDINE	41.9	

Paragon Analytics, Inc.

MDL for Method SW8270

Matrix: LIQUID
Extraction Method: SW3510
AnalysisDate: 02/05/2001
ActiveDate: 05/16/2001
ExpireDate: 02/05/2002
Instrument: HPSV1
Units: UG/L

MDL Comments:
separatory funnel / SW3510.

Analyte:	MDL:	Comments:
BENZO(A)ANTHRACENE	2.34	
BENZO(B)FLUORANTHENE	2.37	
BENZO(B,K)FLUORANTHENE	2.38	
BENZO(G,H,I)PERYLENE	3.59	
BENZO(K)FLUORANTHENE	2.38	
BENZO(A)PYRENE	2.33	
BENZOIC ACID	8.47	
BENZYL ALCOHOL	2.12	
BIS(2-CHLOROETHOXY)METHANE	2.26	
BIS(2-CHLOROETHYL)ETHER	2.1	
BIS(2-CHLOROISOPROPYL)ETHER	2.13	
BIS(2-ETHYLHEXYL)PHTHALATE	2.64	
BUTYL BENZYL PHTHALATE	2.45	
CARBAZOLE	2.64	
CHRYSENE	2.5	
DI-N-BUTYL PHTHALATE	2.52	
DI-N-OCTYL PHTHALATE	2.77	
DIBENZO(A,H)ANTHRACENE	2.66	
DIBENZOFURAN	2.36	
DIETHYL PHTHALATE	4.2	
DIMETHYL PHTHALATE	3.34	
FLUORANTHENE	2.74	
FLUORENE	2.57	
HEXACHLOROBENZENE	2.28	
HEXACHLOROBUTADIENE	1.91	
HEXACHLOROCYCLOPENTADIENE	6.4	
HEXACHLOROETHANE	1.77	
INDENO(1,2,3-CD)PYRENE	3.1	
ISOPHORONE	2.12	
N-NITROSO-DI-N-PROPYLAMINE	2.09	
N-NITROSODIMETHYLAMINE	1.23	
N-NITROSODIPHENYLAMINE	2.27	
NAPHTHALENE	2.27	
NITROBENZENE	2.32	

Paragon Analytics, Inc.

MDL for Method SW8270

Matrix: LIQUID
Extraction Method: SW3510
AnalysisDate: 02/05/2001
ActiveDate: 05/16/2001
ExpireDate: 02/05/2002
Instrument: HPSV1
Units: UG/L

MDL Comments:
separatory funnel / SW3510.

Analyte:	MDL:	Comments:
PENTACHLOROPHENOL	5.39	
PHENANTHRENE	2.42	
PHENOL	0.82	
PYRENE	2.28	
PYRIDINE	1.59	

Paragon Analytics, Inc.

MDL for Method SW8270

Matrix: SOLID
Extraction Method: SW3540
AnalysisDate: 02/05/2001
ActiveDate: 07/18/2001
ExpireDate: 02/05/2002
Instrument: HPSV1
Units: UG/KG

MDL Comments:
solid. 3540/8270.

Analyte:	MDL:	Comments:
1,2,4-TRICHLOROBENZENE	38.9	
1,2-DICHLOROBENZENE	31.6	
1,3-DICHLOROBENZENE	32.4	
1,4-DICHLOROBENZENE	36.6	
2,3,4,6-TETRACHLOROPHENOL	171	
2,4,5-TRICHLOROPHENOL	56.4	
2,4,6-TRICHLOROPHENOL	46.6	
2,4-DICHLOROPHENOL	43.9	
2,4-DIMETHYLPHENOL	48.1	
2,4-DINITROPHENOL	280	
2,4-DINITROTOLUENE	79.5	
2,6-DINITROTOLUENE	49.9	
2-CHLORONAPHTHALENE	42.2	
2-CHLOROPHENOL	30.1	
2-METHYLNAPHTHALENE	53.2	
2-METHYLPHENOL	38.2	
2-NITROANILINE	147	
2-NITROPHENOL	44.5	
3+4-METHYLPHENOL	160	
3,3'-DICHLOROBENZIDINE	334	
3-NITROANILINE	316	
4,6-DINITRO-2-METHYLPHENOL	204	
4-BROMOPHENYL PHENYL ETHER	50.4	
4-CHLORO-3-METHYLPHENOL	67.1	
4-CHLOROANILINE	61.1	
4-CHLOROPHENYL PHENYL ETHER	71.6	
4-METHYLPHENOL	47.7	
4-NITROANILINE	582	
4-NITROPHENOL	496	
ACENAPHTHENE	40.9	
ACENAPHTHYLENE	38	
ANILINE	42.6	
ANTHRACENE	44.3	
AZOBENZENE	55.6	

Paragon Analytics, Inc.

MDL for Method SW8270

Matrix: SOLID
Extraction Method: SW3540
AnalysisDate: 02/05/2001
ActiveDate: 07/18/2001
ExpireDate: 02/05/2002
Instrument: HPSV1
Units: UG/KG

MDL Comments:

solid. 3540/8270.

Analyte:	MDL:	Comments:
BENZIDINE	346	
BENZO(A)ANTHRACENE	30.1	
BENZO(A)PYRENE	36.8	
BENZO(B)FLUORANTHENE	56.6	
BENZO(G,H,I)PERYLENE	138	
BENZO(K)FLUORANTHENE	54.6	
BENZOIC ACID	200	
BENZYL ALCOHOL	53.6	
BIS(2-CHLOROETHOXY)METHANE	46.6	
BIS(2-CHLOROETHYL)ETHER	40.5	
BIS(2-CHLOROISOPROPYL)ETHER	40.4	
BIS(2-ETHYLHEXYL)PHTHALATE	45.2	
BUTYL BENZYL PHTHALATE	42	
CARBAZOLE	76.9	
CHRYSENE	49.3	
DI-N-BUTYL PHTHALATE	42	
DI-N-OCTYL PHTHALATE	104	
DIBENZO(A,H)ANTHRACENE	129	
DIBENZOFURAN	52.5	
DIETHYL PHTHALATE	72.6	
DIMETHYL PHTHALATE	45.9	
FLUORANTHENE	66.9	
FLUORENE	64.2	
HEXACHLOROBENZENE	40.4	
HEXACHLOROBUTADIENE	50.2	
HEXACHLOROCYCLOPENTADIENE	239	
HEXACHLOROETHANE	43.6	
INDENO(1,2,3-CD)PYRENE	133	
ISOPHORONE	48.6	
N-NITROSO-DI-N-PROPYLAMINE	51.3	
N-NITROSODIMETHYLAMINE	55	
N-NITROSODIPHENYLAMINE	50.7	
NAPHTHALENE	41.1	
NITROBENZENE	40.2	

Paragon Analytics, Inc.

MDL for Method SW8270

Matrix: SOLID
Extraction Method: SW3540
AnalysisDate: 02/05/2001
ActiveDate: 07/18/2001
ExpireDate: 02/05/2002
Instrument: HPSV1
Units: UG/KG

MDL Comments:
solid. 3540/8270.

Analyte:	MDL:	Comments:
PENTACHLOROPHENOL	179	
PHENANTHRENE	30	
PHENOL	46.8	
PYRENE	76.5	
PYRIDINE	40.6	

Paragon Analytics, Inc.

MDL for Method SW8270PAH

Matrix: SOLID
Extraction Method: SW3540
AnalysisDate: 02/12/2001
ActiveDate: 02/12/2001
ExpireDate: 02/12/2002
Instrument: HPSV1
Units: UG/KG

MDL Comments:

Entered 2/15/2001. LMP edited 02/23/01 dbh. THESE
MDL VALUES ARE USED FOR "LOW LEVEL" PAHs.

Analyte:	MDL:	Comments:
2-METHYLNAPHTHALENE	13.8	
ACENAPHTHENE	10.7	
ACENAPHTHYLENE	12.1	
ANTHRACENE	13.4	
BENZO(A)ANTHRACENE	12.2	
BENZO(A)PYRENE	16.2	
BENZO(B)FLUORANTHENE	12.2	
BENZO(G,H,I)PERYLENE	19.4	
BENZO(K)FLUORANTHENE	18.4	
CHRYSENE	11.6	
DIBENZO(A,H)ANTHRACENE	30.8	
FLUORANTHENE	14.8	
FLUORENE	16.4	
INDENO(1,2,3-CD)PYRENE	24.8	
NAPHTHALENE	8.31	
PHENANTHRENE	9.48	
PYRENE	13.7	

Paragon Analytics, Inc.

MDL for Method SW8310

Matrix: SOLID
Extraction Method: SW3540
AnalysisDate: 05/11/2001
ActiveDate: 07/02/2001
ExpireDate: 05/11/2002
Instrument: HPLC-1
Units: UG/KG

MDL Comments:

Analyte:	MDL:	Comments:
1-METHYLNAPHTHALENE	8.9	
2-METHYLNAPHTHALENE	11.2	
ACENAPHTHENE	6.17	
ACENAPHTHYLENE	10.4	
ANTHRACENE	0.366	
BENZO(A)ANTHRACENE	0.55	
BENZO(A)PYRENE	0.36	
BENZO(B)FLUORANTHENE	0.492	
BENZO(G,H,I)PERYLENE	0.983	
BENZO(K)FLUORANTHENE	0.473	
CHRYSENE	0.274	
DIBENZO(A,H)ANTHRACENE	0.889	
FLUORANTHENE	0.731	
FLUORENE	1.32	
INDENO(1,2,3-CD)PYRENE	0.36	
NAPHTHALENE	5.36	
PHENANTHRENE	0.557	
PYRENE	0.598	

Paragon Analytics, Inc.

MDL for Method SW8310

Matrix: LIQUID

MDL Comments:

Extraction Method: SW3520

AnalysisDate: 05/11/2001

ActiveDate: 07/02/2001

ExpireDate: 05/11/2002

Instrument: HPLC-1

Units: UG/L

Analyte:	MDL:	Comments:
1-METHYLNAPHTHALENE	0.327	
2-METHYLNAPHTHALENE	0.33	
ACENAPHTHENE	0.174	
ACENAPHTHYLENE	0.323	
ANTHRACENE	0.0108	
BENZO(A)ANTHRACENE	0.0127	
BENZO(A)PYRENE	0.0153	
BENZO(B)FLUORANTHENE	0.0201	
BENZO(G,H,I)PERYLENE	0.028	
BENZO(K)FLUORANTHENE	0.0123	
CHRYSENE	0.00822	
DIBENZO(A,H)ANTHRACENE	0.0216	
FLUORANTHENE	0.0162	
FLUORENE	0.0473	
INDENO(1,2,3-CD)PYRENE	0.013	
NAPHTHALENE	0.128	
PHENANTHRENE	0.0183	
PYRENE	0.0141	

Paragon Analytics, Inc.

MDL for Method SW8330

Matrix: LIQUID
Extraction Method: METHOD
AnalysisDate: 05/08/2001
ActiveDate: 08/02/2001
ExpireDate: 05/08/2002
Instrument: HPLC-1
Units: UG/L

MDL Comments:

C-18 values entered in LIMS. DBH

Analyte:	MDL:	Comments:
1,3,5-TRINITROBENZENE	0.0305	
1,3-DINITROBENZENE	0.03	
2,4,6-TRINITROTOLUENE	0.0175	
2,4-DINITROTOLUENE	0.0243	
2,6-DINITROTOLUENE	0.0292	
2-AMINO-4,6-DNT	0.0301	
2-NITROTOLUENE	0.0181	
3-NITROTOLUENE	0.0301	
4-AMINO-2,6-DNT	0.0297	
4-NITROTOLUENE	0.0291	
HMX	0.0227	
NITROBENZENE	0.0319	
RDX	0.0296	
TETRYL	0.0303	

Paragon Analytics, Inc.

MDL for Method SW8330

Matrix: SOLID
Extraction Method: METHOD
AnalysisDate: 01/09/2001
ActiveDate: 08/06/2001
ExpireDate: 01/09/2002
Instrument: HPLC-1
Units: UG/KG

MDL Comments:

C-18 values entered. DBH

Analyte:	MDL:	Comments:
1,3,5-TRINITROBENZENE	35.2	
1,3-DINITROBENZENE	66.1	
2,4,6-TRINITROTOLUENE	50.9	
2,4-DINITROTOLUENE	59.3	
2,6-DINITROTOLUENE	60.1	
2-AMINO-4,6-DNT	66.4	
2-NITROTOLUENE	47.1	
3-NITROTOLUENE	59.6	
4-AMINO-2,6-DNT	65.3	
4-NITROTOLUENE	53.4	
HMX	57.8	
NITROBENZENE	57.2	
RDX	64.2	
TETRYL	62.6	

Paragon Analytics, Inc.

MDL for Method SW9010

Matrix: SOLID
Extraction Method: METHOD
AnalysisDate: 02/21/2001
ActiveDate: 03/05/2001
ExpireDate: 02/21/2002
Instrument: SPEC
Units: UG/KG

MDL Comments:

Analyte:	MDL:	Comments:
CYANIDE	193	

Paragon Analytics, Inc.

MDL for Method SW9010

Matrix: LIQUID
Extraction Method: METHOD
AnalysisDate: 01/16/2001
ActiveDate: 01/26/2001
ExpireDate: 01/16/2002
Instrument: SPEC
Units: UG/L

MDL Comments:

Analyte:	MDL:	Comments:
CYANIDE	5.28	

ENCLOSURE 4:

Paragon Calibration Data / Case Narratives



Paragon Analytics, Inc.

TOTAL METALS CASE NARRATIVE

Washington Group International, Inc.

EPA RAC -- 49941007

Order Number - 0103075

1. This report consists of six sludge samples.
2. The samples were received cool and intact on 3/10/01.
3. The samples were prepared for analysis based on SW-846, 3rd Edition procedures.

For analysis by Trace ICP, the samples were digested following method 3050B and PAI SOP 806 Rev. 5.

For analysis by Cold Vapor AA (CVAA), the samples were digested following method 7471A and PAI SOP 812 Rev. 7.

4. The samples were analyzed following SW-846 3rd Edition procedures.

Analysis by Trace ICP followed method 6010B and PAI SOP 807 Rev. 5.

The relationship between intensity and concentration for each element is established using at least four standards, one of which is a blank solution. The equation which relates intensity to concentration is:

$$I = A_0 + (A_1 * c^n) + (A_2 * c^{2n})$$

where: I = intensity

c = concentration

A₀ = offset coefficient

A₁ = gain coefficient

A₂ = curvature coefficient

n = exponent coefficient

During sample analysis concentrations are computed by the software and the results are printed in mg/L. The instrument software does not provide a printout which gives both intensity and concentration. The validity of the calibration equation is tested by analyzing the following solutions: a blank, a low level check solution with concentrations near the reporting limit, an Initial

Calibration Verification (ICV) standard from a 2nd source standard solution with concentrations near the middle of the analytical range, a Continuing Calibration Verification (CCV) standard with concentrations at two times those in the ICV, and a readback of the highest calibration standard.

These solutions provide verification that the calibration equations are functioning properly throughout the analytical range of the instrument. During sample analysis dilutions are made for analytes found at concentrations above the highest calibration standard. No results are taken from extrapolations beyond the highest standard.

Analysis by CVAA followed method 7471A and PAI SOP 812 Rev. 7.

The relationship between intensity and concentration is determined daily, prior to sample analysis. At least five standards and a blank solution are analyzed to establish the calibration curve. The instrument software performs a linear regression to fit the calibration data to a curve of the form:

$$\text{conc.} = B * I + C$$

where: conc. = concentration

B = slope coefficient

I = intensity

C = intercept coefficient

A printout summarizing the calibration data supplies the calibration curve and correlation coefficient. During sample analysis both intensity and concentration values are printed. Dilutions are made for concentrations above the highest calibration standard. No results are taken from extrapolations above the highest standard.

5. All standards and solutions are NIST traceable and were used within their recommended shelf life.
6. The samples were prepared and analyzed within the established hold times.

All in house quality control procedures were followed, as described below.

7. General quality control procedures.
 - A preparation (method) blank and laboratory control sample were digested and analyzed with the samples in each digestion batch. There were not more than 20 samples in each digestion batch.
 - The preparation (method) blank results associated with each digestion batch were below the practical quantitation limits for the requested analytes.
 - The laboratory control sample associated with each digestion batch was within the acceptance limits. This indicates complete digestion according to the method.

- All initial and continuing calibration blanks associated with each analytical batch were below the practical quantitation limits for the requested analytes.
- All initial and continuing calibration verifications associated with each analytical batch were within the acceptance criteria for the requested analytes. This indicates a valid calibration and stable instrument conditions.
- The interference check samples and high standard readbacks associated with Method 6010B analyses were within acceptance criteria.

8. Matrix specific quality control procedures.

PAI sample ID 0103168-1 was designated as the quality control sample for the Trace ICP analyses. PAI sample ID 0103075-3 was designated as the quality control sample for the CVAA analysis.

- A matrix spike and matrix spike duplicate were digested and analyzed with each batch. All acceptance criteria for accuracy were met with the following exceptions.

<u>Analyte</u>	<u>Sample ID</u>
Antimony	0103168-1MS and MSD
Manganese	0103168-1MS and MSD

The native sample results are flagged for matrix spike failure and an analytical post spike was performed. Results of the spike were acceptable indicating that the matrix was not significantly affecting quantitation of these analytes.

- Matrix spike recoveries could not be evaluated for the following analytes.

<u>Analyte</u>	<u>Sample ID</u>
Aluminum	0103168-1
Iron	0103168-1

The concentrations of these analytes in the native sample were greater than four times the concentration of matrix spike added during the digestion. When sample concentration is that much greater than the spike added, spike recoveries may not be accurate. The laboratory control sample indicates that the digestion and analysis were in control.

- A sample duplicate and matrix spike duplicate were digested and analyzed with each batch. All acceptance criteria for precision were met.
- A serial dilution was analyzed with the ICP batch. All acceptance criteria were met.

9. PAI sample IDs 0103075-3, -4, -6, and -7 required dilutions to bring lead into the analytical range of the Trace ICP; PAI sample IDs 0103075-6 and -7 required dilutions to bring zinc into analytical range; and PAI sample ID 0103075-8 required a dilution to bring potassium into analytical range.

The data contained in the following report have been reviewed and approved by the personnel listed below:

Melissa Grytdal

Melissa Grytdal

Data Reporting Specialist

4/5/01

Date

SW

Reviewer's Initials

4/9/01

Date

CERTIFICATION

Paragon Analytics, Inc. certifies that the analyses reported herein are true, complete and correct within the limits of the methods employed.

ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: ICV
QC Type: Initial Calibration

Run ID: IT010402-1A1

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	25.3	24.9	0.2		99	90 - 110%
7440-36-0	ANTIMONY	0.25	0.253	0.02		101	90 - 110%
7440-38-2	ARSENIC	0.25	0.257	0.01		103	90 - 110%
7440-39-3	BARIUM	0.25	0.245	0.1		98	90 - 110%
7440-41-7	BERYLLIUM	0.25	0.249	0.005		100	90 - 110%
7440-43-9	CADMIUM	0.25	0.245	0.005		98	90 - 110%
7440-70-2	CALCIUM	25.3	25.1	1		99	90 - 110%
7440-47-3	CHROMIUM	0.25	0.254	0.01		101	90 - 110%
7440-48-4	COBALT	0.25	0.246	0.01		99	90 - 110%
7440-50-8	COPPER	0.25	0.248	0.01		99	90 - 110%
7439-89-6	IRON	10.3	10.2	0.1		99	90 - 110%
7439-92-1	LEAD	0.25	0.258	0.003		103	90 - 110%
7439-95-4	MAGNESIUM	25.3	25.3	1		100	90 - 110%
7439-96-5	MANGANESE	0.25	0.246	0.01		98	90 - 110%
7440-02-0	NICKEL	0.25	0.251	0.02		101	90 - 110%
7440-09-7	POTASSIUM	10	10	1		100	90 - 110%
7782-49-2	SELENIUM	0.25	0.256	0.005		102	90 - 110%
7440-22-4	SILVER	0.25	0.249	0.01		99	90 - 110%
7440-23-5	SODIUM	10.3	10	1		98	90 - 110%
7440-28-0	THALLIUM	0.25	0.249	0.01		100	90 - 110%
7440-62-2	VANADIUM	0.25	0.246	0.01		98	90 - 110%
7440-66-6	ZINC	0.25	0.249	0.02		100	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

Paragon Analytics Inc.
LIMS Version: 1.935

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

Client/Project ID: EPA RAC 49941007

Lab ID: CCV1
QC Type: Continuing Calibration

Run ID: IT010402-1A1
Date Analyzed: 04/02/2001
Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.9	0.2		99	90 - 110%
7440-36-0	ANTIMONY	0.5	0.508	0.02		102	90 - 110%
7440-38-2	ARSENIC	0.5	0.508	0.01		102	90 - 110%
7440-39-3	BARIUM	0.5	0.493	0.1		99	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.495	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.483	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	50.5	1		100	90 - 110%
7440-47-3	CHROMIUM	0.5	0.503	0.01		101	90 - 110%
7440-48-4	COBALT	0.5	0.486	0.01		97	90 - 110%
7440-50-8	COPPER	0.5	0.503	0.01		101	90 - 110%
7439-89-6	IRON	20.5	20.4	0.1		100	90 - 110%
7439-92-1	LEAD	0.5	0.51	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	50.6	1		100	90 - 110%
7439-96-5	MANGANESE	0.5	0.487	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.534	0.02		107	90 - 110%
7440-09-7	POTASSIUM	20	20.2	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.505	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.499	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.5	1		100	90 - 110%
7440-28-0	THALLIUM	0.5	0.497	0.01		99	90 - 110%
7440-62-2	VANADIUM	0.5	0.49	0.01		98	90 - 110%
7440-66-6	ZINC	0.5	0.494	0.02		99	90 - 110%

Data Package ID: IT0103075-1

ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV2

QC Type: Continuing Calibration

Run ID: IT010402-1A1

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.7	0.2		99	90 - 110%
7440-36-0	ANTIMONY	0.5	0.503	0.02		101	90 - 110%
7440-38-2	ARSENIC	0.5	0.506	0.01		101	90 - 110%
7440-39-3	BARIUM	0.5	0.494	0.1		99	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.494	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.484	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	50.5	1		100	90 - 110%
7440-47-3	CHROMIUM	0.5	0.502	0.01		100	90 - 110%
7440-48-4	COBALT	0.5	0.486	0.01		97	90 - 110%
7440-50-8	COPPER	0.5	0.502	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20.4	0.1		99	90 - 110%
7439-92-1	LEAD	0.5	0.512	0.003		103	90 - 110%
7439-95-4	MAGNESIUM	50.5	50.6	1		100	90 - 110%
7439-96-5	MANGANESE	0.5	0.487	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.535	0.02		107	90 - 110%
7440-09-7	POTASSIUM	20	20.2	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.507	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.501	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.5	1		100	90 - 110%
7440-28-0	THALLIUM	0.5	0.5	0.01		100	90 - 110%
7440-62-2	VANADIUM	0.5	0.488	0.01		98	90 - 110%
7440-66-6	ZINC	0.5	0.494	0.02		99	90 - 110%

Data Package ID: IT0103075-1

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Paragon Analytics Inc.

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV3
QC Type: Continuing Calibration

Run ID: IT010402-1A1

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.5	0.2		98	90 - 110%
7440-36-0	ANTIMONY	0.5	0.507	0.02		101	90 - 110%
7440-38-2	ARSENIC	0.5	0.507	0.01		101	90 - 110%
7440-39-3	BARIUM	0.5	0.491	0.1		98	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.493	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.486	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	50.4	1		100	90 - 110%
7440-47-3	CHROMIUM	0.5	0.503	0.01		101	90 - 110%
7440-48-4	COBALT	0.5	0.485	0.01		97	90 - 110%
7440-50-8	COPPER	0.5	0.498	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20.3	0.1		99	90 - 110%
7439-92-1	LEAD	0.5	0.513	0.003		103	90 - 110%
7439-95-4	MAGNESIUM	50.5	50.5	1		100	90 - 110%
7439-96-5	MANGANESE	0.5	0.486	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.538	0.02		108	90 - 110%
7440-09-7	POTASSIUM	20	20.2	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.509	0.005		102	90 - 110%
7440-22-4	SILVER	0.5	0.501	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.6	1		101	90 - 110%
7440-28-0	THALLIUM	0.5	0.501	0.01		100	90 - 110%
7440-62-2	VANADIUM	0.5	0.487	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.492	0.02		98	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

Paragon Analytics Inc.

LIMS Version: 1.935

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV4

QC Type: Continuing Calibration

Run ID: IT010402-1A1

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.3	0.2		98	90 - 110%
7440-36-0	ANTIMONY	0.5	0.502	0.02		101	90 - 110%
7440-38-2	ARSENIC	0.5	0.503	0.01		101	90 - 110%
7440-39-3	BARIUM	0.5	0.489	0.1		98	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.491	0.005		98	90 - 110%
7440-43-9	CADMIUM	0.5	0.484	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	50.2	1		100	90 - 110%
7440-47-3	CHROMIUM	0.5	0.5	0.01		100	90 - 110%
7440-48-4	COBALT	0.5	0.483	0.01		97	90 - 110%
7440-50-8	COPPER	0.5	0.495	0.01		99	90 - 110%
7439-89-6	IRON	20.5	20.3	0.1		99	90 - 110%
7439-92-1	LEAD	0.5	0.512	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	50.3	1		100	90 - 110%
7439-96-5	MANGANESE	0.5	0.484	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.536	0.02		107	90 - 110%
7440-09-7	POTASSIUM	20	20.1	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.507	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.499	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.5	1		100	90 - 110%
7440-28-0	THALLIUM	0.5	0.493	0.01		99	90 - 110%
7440-62-2	VANADIUM	0.5	0.485	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.489	0.02		98	90 - 110%

Data Package ID: IT0103075-1

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV5
QC Type: Continuing Calibration

Run ID: IT010402-1A1
Date Analyzed: 04/02/2001
Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.1	0.2		97	90 - 110%
7440-36-0	ANTIMONY	0.5	0.507	0.02		101	90 - 110%
7440-38-2	ARSENIC	0.5	0.504	0.01		101	90 - 110%
7440-39-3	BARIUM	0.5	0.49	0.1		98	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.489	0.005		98	90 - 110%
7440-43-9	CADMIUM	0.5	0.481	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.495	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.482	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.495	0.01		99	90 - 110%
7439-89-6	IRON	20.5	20.2	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.51	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	50.2	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.475	0.01		95	90 - 110%
7440-02-0	NICKEL	0.5	0.517	0.02		103	90 - 110%
7440-09-7	POTASSIUM	20	20.2	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.508	0.005		102	90 - 110%
7440-22-4	SILVER	0.5	0.499	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.2	1		99	90 - 110%
7440-28-0	THALLIUM	0.5	0.496	0.01		99	90 - 110%
7440-62-2	VANADIUM	0.5	0.484	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.484	0.02		97	90 - 110%

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV6

QC Type: Continuing Calibration

Run ID: IT010402-1A1

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	48.7	0.2		97	90 - 110%
7440-36-0	ANTIMONY	0.5	0.502	0.02		100	90 - 110%
7440-38-2	ARSENIC	0.5	0.498	0.01		100	90 - 110%
7440-39-3	BARIUM	0.5	0.482	0.1		96	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.486	0.005		97	90 - 110%
7440-43-9	CADMIUM	0.5	0.476	0.005		95	90 - 110%
7440-70-2	CALCIUM	50.5	49.6	1		98	90 - 110%
7440-47-3	CHROMIUM	0.5	0.493	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.479	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.49	0.01		98	90 - 110%
7439-89-6	IRON	20.5	20	0.1		97	90 - 110%
7439-92-1	LEAD	0.5	0.505	0.003		101	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.9	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.473	0.01		95	90 - 110%
7440-02-0	NICKEL	0.5	0.51	0.02		102	90 - 110%
7440-09-7	POTASSIUM	20	20	1		100	90 - 110%
7782-49-2	SELENIUM	0.5	0.498	0.005		100	90 - 110%
7440-22-4	SILVER	0.5	0.495	0.01		99	90 - 110%
7440-23-5	SODIUM	20.5	20.1	1		98	90 - 110%
7440-28-0	THALLIUM	0.5	0.491	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.48	0.01		96	90 - 110%
7440-66-6	ZINC	0.5	0.477	0.02		95	90 - 110%

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV7
QC Type: Continuing Calibration

Run ID: IT010402-1A1
Date Analyzed: 04/02/2001
Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	48.3	0.2		96	90 - 110%
7440-36-0	ANTIMONY	0.5	0.494	0.02		99	90 - 110%
7440-38-2	ARSENIC	0.5	0.495	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.475	0.1		95	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.485	0.005		97	90 - 110%
7440-43-9	CADMIUM	0.5	0.492	0.005		98	90 - 110%
7440-70-2	CALCIUM	50.5	49.8	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.494	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.478	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.484	0.01		97	90 - 110%
7439-89-6	IRON	20.5	20	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.509	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.9	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.487	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.512	0.02		102	90 - 110%
7440-09-7	POTASSIUM	20	19.8	1		99	90 - 110%
7782-49-2	SELENIUM	0.5	0.5	0.005		100	90 - 110%
7440-22-4	SILVER	0.5	0.493	0.01		99	90 - 110%
7440-23-5	SODIUM	20.5	20.1	1		98	90 - 110%
7440-28-0	THALLIUM	0.5	0.491	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.478	0.01		96	90 - 110%
7440-66-6	ZINC	0.5	0.494	0.02		99	90 - 110%

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

Client/Project ID: EPA RAC 49941007

Lab ID: CCV8

Run ID: IT010402-1A1

QC Type: Continuing Calibration

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	48.9	0.2		97	90 - 110%
7440-36-0	ANTIMONY	0.5	0.502	0.02		100	90 - 110%
7440-38-2	ARSENIC	0.5	0.505	0.01		101	90 - 110%
7440-39-3	BARIUM	0.5	0.483	0.1		97	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.488	0.005		98	90 - 110%
7440-43-9	CADMIUM	0.5	0.496	0.005		99	90 - 110%
7440-70-2	CALCIUM	50.5	50.2	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.498	0.01		100	90 - 110%
7440-48-4	COBALT	0.5	0.482	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.491	0.01		98	90 - 110%
7439-89-6	IRON	20.5	20.1	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.511	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	50.2	1		100	90 - 110%
7439-96-5	MANGANESE	0.5	0.492	0.01		98	90 - 110%
7440-02-0	NICKEL	0.5	0.514	0.02		103	90 - 110%
7440-09-7	POTASSIUM	20	20	1		100	90 - 110%
7782-49-2	SELENIUM	0.5	0.504	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.498	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.4	1		100	90 - 110%
7440-28-0	THALLIUM	0.5	0.497	0.01		99	90 - 110%
7440-62-2	VANADIUM	0.5	0.482	0.01		96	90 - 110%
7440-66-6	ZINC	0.5	0.491	0.02		98	90 - 110%

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV9

QC Type: Continuing Calibration

Run ID: IT010402-1A1

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	48.9	0.2		97	90 - 110%
7440-36-0	ANTIMONY	0.5	0.492	0.02		98	90 - 110%
7440-38-2	ARSENIC	0.5	0.493	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.477	0.1		96	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.485	0.005		97	90 - 110%
7440-43-9	CADMIUM	0.5	0.487	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	50.1	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.494	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.475	0.01		95	90 - 110%
7440-50-8	COPPER	0.5	0.484	0.01		97	90 - 110%
7439-89-6	IRON	20.5	20	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.506	0.003		101	90 - 110%
7439-95-4	MAGNESIUM	50.5	50	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.489	0.01		98	90 - 110%
7440-02-0	NICKEL	0.5	0.504	0.02		101	90 - 110%
7440-09-7	POTASSIUM	20	19.8	1		99	90 - 110%
7782-49-2	SELENIUM	0.5	0.494	0.005		99	90 - 110%
7440-22-4	SILVER	0.5	0.492	0.01		99	90 - 110%
7440-23-5	SODIUM	20.5	20.1	1		98	90 - 110%
7440-28-0	THALLIUM	0.5	0.488	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.478	0.01		96	90 - 110%
7440-66-6	ZINC	0.5	0.494	0.02		99	90 - 110%

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV10

QC Type: Continuing Calibration

Run ID: IT010402-1A1

Date Analyzed: 04/02/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	48.4	0.2		96	90 - 110%
7440-36-0	ANTIMONY	0.5	0.492	0.02		98	90 - 110%
7440-38-2	ARSENIC	0.5	0.494	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.476	0.1		95	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.478	0.005		96	90 - 110%
7440-43-9	CADMIUM	0.5	0.483	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	49.3	1		98	90 - 110%
7440-47-3	CHROMIUM	0.5	0.487	0.01		97	90 - 110%
7440-48-4	COBALT	0.5	0.47	0.01		94	90 - 110%
7440-50-8	COPPER	0.5	0.479	0.01		96	90 - 110%
7439-89-6	IRON	20.5	19.8	0.1		97	90 - 110%
7439-92-1	LEAD	0.5	0.498	0.003		100	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.4	1		98	90 - 110%
7439-96-5	MANGANESE	0.5	0.483	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.494	0.02		99	90 - 110%
7440-09-7	POTASSIUM	20	19.7	1		98	90 - 110%
7782-49-2	SELENIUM	0.5	0.499	0.005		100	90 - 110%
7440-22-4	SILVER	0.5	0.487	0.01		98	90 - 110%
7440-23-5	SODIUM	20.5	20	1		98	90 - 110%
7440-28-0	THALLIUM	0.5	0.487	0.01		97	90 - 110%
7440-62-2	VANADIUM	0.5	0.471	0.01		94	90 - 110%
7440-66-6	ZINC	0.5	0.479	0.02		96	90 - 110%

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV11
QC Type: Continuing Calibration

Run ID: IT010402-1A1
Date Analyzed: 04/02/2001
Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	48.2	0.2		96	90 - 110%
7440-36-0	ANTIMONY	0.5	0.489	0.02		98	90 - 110%
7440-38-2	ARSENIC	0.5	0.491	0.01		98	90 - 110%
7440-39-3	BARIUM	0.5	0.478	0.1		96	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.475	0.005		95	90 - 110%
7440-43-9	CADMIUM	0.5	0.483	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	49.3	1		98	90 - 110%
7440-47-3	CHROMIUM	0.5	0.488	0.01		98	90 - 110%
7440-48-4	COBALT	0.5	0.469	0.01		94	90 - 110%
7440-50-8	COPPER	0.5	0.478	0.01		96	90 - 110%
7439-89-6	IRON	20.5	19.8	0.1		96	90 - 110%
7439-92-1	LEAD	0.5	0.498	0.003		100	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.2	1		98	90 - 110%
7439-96-5	MANGANESE	0.5	0.482	0.01		96	90 - 110%
7440-02-0	NICKEL	0.5	0.494	0.02		99	90 - 110%
7440-09-7	POTASSIUM	20	19.7	1		98	90 - 110%
7782-49-2	SELENIUM	0.5	0.5	0.005		100	90 - 110%
7440-22-4	SILVER	0.5	0.488	0.01		98	90 - 110%
7440-23-5	SODIUM	20.5	20.1	1		98	90 - 110%
7440-28-0	THALLIUM	0.5	0.485	0.01		97	90 - 110%
7440-62-2	VANADIUM	0.5	0.472	0.01		94	90 - 110%
7440-66-6	ZINC	0.5	0.477	0.02		95	90 - 110%

Data Package ID: IT0103075-1

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: ICV
QC Type: Initial Calibration

Run ID: IT010403-1A1
Date Analyzed: 04/03/2001
Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	25.3	24.6	0.2		97	90 - 110%
7440-36-0	ANTIMONY	0.25	0.246	0.02		98	90 - 110%
7440-38-2	ARSENIC	0.25	0.246	0.01		98	90 - 110%
7440-39-3	BARIUM	0.25	0.246	0.1		98	90 - 110%
7440-41-7	BERYLLIUM	0.25	0.249	0.005		100	90 - 110%
7440-43-9	CADMIUM	0.25	0.244	0.005		98	90 - 110%
7440-70-2	CALCIUM	25.3	24.7	1		98	90 - 110%
7440-47-3	CHROMIUM	0.25	0.252	0.01		101	90 - 110%
7440-48-4	COBALT	0.25	0.244	0.01		98	90 - 110%
7440-50-8	COPPER	0.25	0.246	0.01		98	90 - 110%
7439-89-6	IRON	10.3	9.91	0.1		97	90 - 110%
7439-92-1	LEAD	0.25	0.253	0.003		101	90 - 110%
7439-95-4	MAGNESIUM	25.3	24.8	1		98	90 - 110%
7439-96-5	MANGANESE	0.25	0.244	0.01		98	90 - 110%
7440-02-0	NICKEL	0.25	0.248	0.02		99	90 - 110%
7440-09-7	POTASSIUM	10	10.1	1		101	90 - 110%
7782-49-2	SELENIUM	0.25	0.252	0.005		101	90 - 110%
7440-22-4	SILVER	0.25	0.246	0.01		99	90 - 110%
7440-23-5	SODIUM	10.3	9.92	1		97	90 - 110%
7440-28-0	THALLIUM	0.25	0.243	0.01		97	90 - 110%
7440-62-2	VANADIUM	0.25	0.245	0.01		98	90 - 110%
7440-66-6	ZINC	0.25	0.258	0.02		103	90 - 110%

Data Package ID: IT0103075-1

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV1
QC Type: Continuing Calibration

Run ID: IT010403-1A1
Date Analyzed: 04/03/2001
Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	50.2	0.2		99	90 - 110%
7440-36-0	ANTIMONY	0.5	0.5	0.02		100	90 - 110%
7440-38-2	ARSENIC	0.5	0.501	0.01		100	90 - 110%
7440-39-3	BARIUM	0.5	0.5	0.1		100	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.499	0.005		100	90 - 110%
7440-43-9	CADMIUM	0.5	0.485	0.005		97	90 - 110%
7440-70-2	CALCIUM	50.5	50.6	1		100	90 - 110%
7440-47-3	CHROMIUM	0.5	0.503	0.01		101	90 - 110%
7440-48-4	COBALT	0.5	0.487	0.01		98	90 - 110%
7440-50-8	COPPER	0.5	0.505	0.01		101	90 - 110%
7439-89-6	IRON	20.5	20.3	0.1		99	90 - 110%
7439-92-1	LEAD	0.5	0.509	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	50.4	1		100	90 - 110%
7439-96-5	MANGANESE	0.5	0.49	0.01		98	90 - 110%
7440-02-0	NICKEL	0.5	0.526	0.02		105	90 - 110%
7440-09-7	POTASSIUM	20	20.6	1		103	90 - 110%
7782-49-2	SELENIUM	0.5	0.504	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.499	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.7	1		101	90 - 110%
7440-28-0	THALLIUM	0.5	0.495	0.01		99	90 - 110%
7440-62-2	VANADIUM	0.5	0.494	0.01		99	90 - 110%
7440-66-6	ZINC	0.5	0.488	0.02		98	90 - 110%

Data Package ID: IT0103075-1

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV2

QC Type: Continuing Calibration

Run ID: IT010403-1A1

Date Analyzed: 04/03/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.7	0.2		98	90 - 110%
7440-36-0	ANTIMONY	0.5	0.498	0.02		100	90 - 110%
7440-38-2	ARSENIC	0.5	0.496	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.495	0.1		99	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.496	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.481	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50.1	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.498	0.01		100	90 - 110%
7440-48-4	COBALT	0.5	0.482	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.5	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20.2	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.509	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.9	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.485	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.512	0.02		102	90 - 110%
7440-09-7	POTASSIUM	20	20.2	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.499	0.005		100	90 - 110%
7440-22-4	SILVER	0.5	0.497	0.01		99	90 - 110%
7440-23-5	SODIUM	20.5	20.6	1		101	90 - 110%
7440-28-0	THALLIUM	0.5	0.489	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.487	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.497	0.02		100	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV3

QC Type: Continuing Calibration

Run ID: IT010403-1A1

Date Analyzed: 04/03/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.6	0.2		98	90 - 110%
7440-36-0	ANTIMONY	0.5	0.499	0.02		100	90 - 110%
7440-38-2	ARSENIC	0.5	0.497	0.01		100	90 - 110%
7440-39-3	BARIUM	0.5	0.497	0.1		99	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.493	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.481	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.497	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.481	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.499	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.508	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.7	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.484	0.01		97	90 - 110%
7440-02-0	NICKEL	0.5	0.512	0.02		102	90 - 110%
7440-09-7	POTASSIUM	20	20.3	1		102	90 - 110%
7782-49-2	SELENIUM	0.5	0.506	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.497	0.01		99	90 - 110%
7440-23-5	SODIUM	20.5	20.7	1		101	90 - 110%
7440-28-0	THALLIUM	0.5	0.487	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.486	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.479	0.02		96	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV4

QC Type: Continuing Calibration

Run ID: IT010403-1A1

Date Analyzed: 04/03/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.6	0.2		98	90 - 110%
7440-36-0	ANTIMONY	0.5	0.494	0.02		99	90 - 110%
7440-38-2	ARSENIC	0.5	0.495	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.492	0.1		99	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.494	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.48	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50.1	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.496	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.483	0.01		97	90 - 110%
7440-50-8	COPPER	0.5	0.498	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20.1	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.509	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.8	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.481	0.01		96	90 - 110%
7440-02-0	NICKEL	0.5	0.512	0.02		102	90 - 110%
7440-09-7	POTASSIUM	20	20.2	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.501	0.005		100	90 - 110%
7440-22-4	SILVER	0.5	0.497	0.01		99	90 - 110%
7440-23-5	SODIUM	20.5	20.7	1		101	90 - 110%
7440-28-0	THALLIUM	0.5	0.486	0.01		97	90 - 110%
7440-62-2	VANADIUM	0.5	0.486	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.478	0.02		96	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV5

QC Type: Continuing Calibration

Run ID: IT010403-1A1

Date Analyzed: 04/03/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.8	0.2		99	90 - 110%
7440-36-0	ANTIMONY	0.5	0.497	0.02		99	90 - 110%
7440-38-2	ARSENIC	0.5	0.498	0.01		100	90 - 110%
7440-39-3	BARIUM	0.5	0.494	0.1		99	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.494	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.481	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50.3	1		100	90 - 110%
7440-47-3	CHROMIUM	0.5	0.498	0.01		100	90 - 110%
7440-48-4	COBALT	0.5	0.481	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.5	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20.2	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.507	0.003		101	90 - 110%
7439-95-4	MAGNESIUM	50.5	50	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.482	0.01		96	90 - 110%
7440-02-0	NICKEL	0.5	0.506	0.02		101	90 - 110%
7440-09-7	POTASSIUM	20	20.3	1		102	90 - 110%
7782-49-2	SELENIUM	0.5	0.504	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.5	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.8	1		102	90 - 110%
7440-28-0	THALLIUM	0.5	0.49	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.487	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.473	0.02		95	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV6

QC Type: Continuing Calibration

Run ID: IT010403-1A1

Date Analyzed: 04/03/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.9	0.2		99	90 - 110%
7440-36-0	ANTIMONY	0.5	0.496	0.02		99	90 - 110%
7440-38-2	ARSENIC	0.5	0.495	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.494	0.1		99	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.494	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.48	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50.1	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.497	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.481	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.5	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20.1	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.506	0.003		101	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.9	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.482	0.01		96	90 - 110%
7440-02-0	NICKEL	0.5	0.504	0.02		101	90 - 110%
7440-09-7	POTASSIUM	20	20.3	1		102	90 - 110%
7782-49-2	SELENIUM	0.5	0.505	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.5	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.8	1		102	90 - 110%
7440-28-0	THALLIUM	0.5	0.488	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.486	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.486	0.02		97	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV7

QC Type: Continuing Calibration

Run ID: IT010403-1A1

Date Analyzed: 04/03/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.8	0.2		99	90 - 110%
7440-36-0	ANTIMONY	0.5	0.499	0.02		100	90 - 110%
7440-38-2	ARSENIC	0.5	0.495	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.492	0.1		98	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.493	0.005		99	90 - 110%
7440-43-9	CADMIUM	0.5	0.481	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50.3	1		100	90 - 110%
7440-47-3	CHROMIUM	0.5	0.497	0.01		100	90 - 110%
7440-48-4	COBALT	0.5	0.481	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.498	0.01		100	90 - 110%
7439-89-6	IRON	20.5	20.1	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.508	0.003		102	90 - 110%
7439-95-4	MAGNESIUM	50.5	50	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.48	0.01		96	90 - 110%
7440-02-0	NICKEL	0.5	0.507	0.02		101	90 - 110%
7440-09-7	POTASSIUM	20	20.3	1		102	90 - 110%
7782-49-2	SELENIUM	0.5	0.505	0.005		101	90 - 110%
7440-22-4	SILVER	0.5	0.5	0.01		100	90 - 110%
7440-23-5	SODIUM	20.5	20.8	1		102	90 - 110%
7440-28-0	THALLIUM	0.5	0.492	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.486	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.484	0.02		97	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

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ICP Metals

Method SW6010

Calibration Verifications

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Lab ID: CCV8

QC Type: Continuing Calibration

Run ID: IT010403-1A1

Date Analyzed: 04/03/2001

Result Units: MG/L

CASNO	Target Analyte	Spike Added	Result	Reporting Limit	Result Qualifier	% Rec.	Control Limits
7429-90-5	ALUMINUM	50.5	49.6	0.2		98	90 - 110%
7440-36-0	ANTIMONY	0.5	0.496	0.02		99	90 - 110%
7440-38-2	ARSENIC	0.5	0.497	0.01		99	90 - 110%
7440-39-3	BARIUM	0.5	0.492	0.1		98	90 - 110%
7440-41-7	BERYLLIUM	0.5	0.49	0.005		98	90 - 110%
7440-43-9	CADMIUM	0.5	0.478	0.005		96	90 - 110%
7440-70-2	CALCIUM	50.5	50	1		99	90 - 110%
7440-47-3	CHROMIUM	0.5	0.495	0.01		99	90 - 110%
7440-48-4	COBALT	0.5	0.477	0.01		96	90 - 110%
7440-50-8	COPPER	0.5	0.497	0.01		99	90 - 110%
7439-89-6	IRON	20.5	20	0.1		98	90 - 110%
7439-92-1	LEAD	0.5	0.504	0.003		101	90 - 110%
7439-95-4	MAGNESIUM	50.5	49.7	1		99	90 - 110%
7439-96-5	MANGANESE	0.5	0.478	0.01		96	90 - 110%
7440-02-0	NICKEL	0.5	0.502	0.02		101	90 - 110%
7440-09-7	POTASSIUM	20	20.3	1		101	90 - 110%
7782-49-2	SELENIUM	0.5	0.498	0.005		100	90 - 110%
7440-22-4	SILVER	0.5	0.497	0.01		99	90 - 110%
7440-23-5	SODIUM	20.5	20.8	1		101	90 - 110%
7440-28-0	THALLIUM	0.5	0.488	0.01		98	90 - 110%
7440-62-2	VANADIUM	0.5	0.484	0.01		97	90 - 110%
7440-66-6	ZINC	0.5	0.479	0.02		96	90 - 110%

Data Package ID: IT0103075-1

Date Printed: Thursday, April 05, 2001

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Metals Linear Ranges

Lab Name: Paragon Analytics, Inc.

Work Order Number: 0103075

Client Name: Washington Group International, Inc.

ClientProject ID: EPA RAC 49941007

Instrument ID: ICPTrace

Active Date: 01/16/2001

Expiration Date: 04/15/2001

CASNO	Target Analyte	Concentration (ppm)
7429-90-5	Aluminum	500
7440-36-0	Antimony	2
7440-38-2	Arsenic	10
7440-39-3	Barium	10
7440-41-7	Beryllium	10
7440-43-9	Cadmium	10
7440-70-2	Calcium	500
7440-47-3	Chromium	10
7440-48-4	Cobalt	10
7440-50-8	Copper	10
7439-89-6	Iron	200
7439-92-1	Lead	10
7439-95-4	Magnesium	500
7439-96-5	Manganese	10
7440-02-0	Nickel	10
7440-09-7	Potassium	100
7782-49-2	Selenium	10
7440-22-4	Silver	2
7440-23-5	Sodium	100
7440-28-0	Thallium	10
7440-62-2	Vanadium	10
7440-66-6	Zinc	10

ICP Run Log -- 4/2/2001

Instrument ID: ICPTrace
 File Name: TS10402
 AnalRunID: IT010402-1A1
 CalibRefID: IT010402-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
		MIXBHGH	MIXBH	1	4/2/01	09:26	
		MIXAHGH	MIXAH	1	4/2/01	09:28	
		ICV	ICV	1	4/2/01	09:39	
		ICB	ICB	1	4/2/01	09:42	
		CRI1	CRI	1	4/2/01	09:45	
		ICSA1	ICSA	1	4/2/01	09:49	
		ICSAB1	ICSAB	1	4/2/01	09:51	
		CCV1	CCV	1	4/2/01	09:54	
		CCB1	CCB	1	4/2/01	09:56	
		IP010330-1	MB	1	4/2/01	10:00	IP010330-1
		IP010330-1	LCS	1	4/2/01	10:02	IP010330-1
		IP010330-1	LCSD	1	4/2/01	10:05	IP010330-1
		0103190-1	SMP	1	4/2/01	10:07	IP010330-1
		0103190-2	SMP	1	4/2/01	10:09	IP010330-1
		0103190-3	SMP	1	4/2/01	10:12	IP010330-1
		CRI2	CRI	1	4/2/01	10:14	
		ICSA2	ICSA	1	4/2/01	10:16	
		ICSAB2	ICSAB	1	4/2/01	10:19	
		CCV2	CCV	1	4/2/01	10:21	
		CCB2	CCB	1	4/2/01	10:24	
		IP010330-2	MB	1	4/2/01	10:30	IP010330-2
		IP010330-2	LCS	1	4/2/01	10:33	IP010330-2
		0103174-2	SMP	1	4/2/01	10:35	IP010330-2
		0103174-2	DUP	1	4/2/01	10:37	IP010330-2
		0103174-2	SER	5	4/2/01	10:40	IP010330-2
		0103174-2	MS	1	4/2/01	10:42	IP010330-2
		0103174-2	MSD	1	4/2/01	10:44	IP010330-2
		0103174-3	SMP	1	4/2/01	10:47	IP010330-2
		0103174-4	SMP	1	4/2/01	10:49	IP010330-2
		0103174-5	SMP	1	4/2/01	10:51	IP010330-2
		CCV3	CCV	1	4/2/01	10:54	
		CCB3	CCB	1	4/2/01	10:56	
		0103174-6	SMP	1	4/2/01	10:59	IP010330-2
		0103174-7	SMP	1	4/2/01	11:01	IP010330-2
		0103174-8	SMP	1	4/2/01	11:03	IP010330-2
		0103174-9	SMP	1	4/2/01	11:06	IP010330-2
		0103174-10	SMP	1	4/2/01	11:08	IP010330-2

Data Package ID: IT0103075-1

ICP Run Log -- 4/2/2001

Instrument ID: ICPTrace

File Name: TS10402

AnalRunID: IT010402-1A1

CalibRefID: IT010402-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
		0103174-11	SMP	1	4/2/01	11:11	IP010330-2
		0103174-12	SMP	1	4/2/01	11:13	IP010330-2
		0103174-13	SMP	1	4/2/01	11:15	IP010330-2
		0103174-14	SMP	1	4/2/01	11:18	IP010330-2
		0103174-2	SER	5	4/2/01	11:21	IP010330-2
		CCV4	CCV	1	4/2/01	11:24	
		CCB4	CCB	1	4/2/01	11:26	
		0103163-11	SMP	1	4/2/01	11:28	IP010330-2
		0103165-4	SMP	1	4/2/01	11:31	IP010330-2
		0103165-5	SMP	1	4/2/01	11:33	IP010330-2
		0103165-4	SMP	2	4/2/01	11:38	IP010330-2
		0103165-5	SMP	10	4/2/01	11:40	IP010330-2
		0103165-5	SMP	100	4/2/01	11:43	IP010330-2
		CRI3	CRI	1	4/2/01	11:45	
		ICSA3	ICSA	1	4/2/01	11:48	
		ICSAB3	ICSAB	1	4/2/01	11:50	
		CCV5	CCV	1	4/2/01	11:52	
		CCB5	CCB	1	4/2/01	11:55	
		IP010330-3	MB	1	4/2/01	12:01	IP010330-3
		IP010330-3	LCS	1	4/2/01	12:03	IP010330-3
		0103168-1	SMP	1	4/2/01	12:05	IP010330-3
		0103168-1	DUP	1	4/2/01	12:08	IP010330-3
		0103168-1	SER	5	4/2/01	12:10	IP010330-3
		0103168-1	MS	1	4/2/01	12:13	IP010330-3
		0103168-1	MSD	1	4/2/01	12:15	IP010330-3
		0103163-1	SMP	1	4/2/01	12:17	IP010330-3
		0103163-2	SMP	1	4/2/01	12:20	IP010330-3
		0103163-3	SMP	1	4/2/01	12:22	IP010330-3
		CCV6	CCV	1	4/2/01	12:24	
		CCB6	CCB	1	4/2/01	12:27	
		0103163-4	SMP	1	4/2/01	12:31	IP010330-3
		0103163-5	SMP	1	4/2/01	12:33	IP010330-3
		0103163-6	SMP	1	4/2/01	12:36	IP010330-3
		0103163-7	SMP	1	4/2/01	12:38	IP010330-3
		0103163-8	SMP	1	4/2/01	12:41	IP010330-3
		0103163-9	SMP	1	4/2/01	12:43	IP010330-3
		0103163-10	SMP	1	4/2/01	12:46	IP010330-3

Data Package ID: IT0103075-1

ICP Run Log -- 4/2/2001

Instrument ID: ICPTrace
 File Name: TS10402
 AnalRunID: IT010402-1A1
 CalibRefID: IT010402-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
	T-01-S	0103075-3	SMP	1	4/2/01	12:48	IP010330-3
	T-02-S	0103075-4	SMP	1	4/2/01	12:51	IP010330-3
	D-01-S	0103075-6	SMP	1	4/2/01	12:54	IP010330-3
		CCV7	CCV	1	4/2/01	12:56	
		CCB7	CCB	1	4/2/01	12:59	
	D-02-S	0103075-7	SMP	1	4/2/01	13:01	IP010330-3
	D-03-S	0103075-8	SMP	1	4/2/01	13:04	IP010330-3
	D-04-S	0103075-9	SMP	1	4/2/01	13:06	IP010330-3
		0103168-1	A	1	4/2/01	13:09	IP010330-3
		CCV8	CCV	1	4/2/01	13:12	
		CCB8	CCB	1	4/2/01	13:14	
		CCV9	CCV	1	4/2/01	14:37	
		CCB9	CCB	1	4/2/01	14:40	
	T-02-S	0103075-4	SMP	10	4/2/01	14:42	IP010330-3
	D-01-S	0103075-6	SMP	10	4/2/01	14:45	IP010330-3
	D-02-S	0103075-7	SMP	10	4/2/01	14:47	IP010330-3
	T-01-S	0103075-3	SMP	10	4/2/01	14:51	IP010330-3
	D-03-S	0103075-8	SMP	1	4/2/01	14:53	IP010330-3
		CCV10	CCV	1	4/2/01	14:57	
		CCB10	CCB	1	4/2/01	15:01	
		IDL-1	IDL-1	1	4/2/01	15:04	
		IDL-2	IDL-2	1	4/2/01	15:07	
		IDL-3	IDL-3	1	4/2/01	15:09	
		IDL-4	IDL-4	1	4/2/01	15:11	
		IDL-5	IDL-5	1	4/2/01	15:14	
		IDL-6	IDL-6	1	4/2/01	15:16	
		IDL-7	IDL-7	1	4/2/01	15:18	
		CRI4	CRI	1	4/2/01	15:22	
		ICSA4	ICSA	1	4/2/01	15:24	
		ICSAB4	ICSAB	1	4/2/01	15:27	
		CCV11	CCV	1	4/2/01	15:29	
		CCB11	CCB	1	4/2/01	15:32	

Data Package ID: IT0103075-1

ICP Run Log -- 4/3/2001

Instrument ID: ICPTrace
 File Name: TS10403
 AnalRunID: IT010403-1A1
 CalibRefID: IT010403-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
		MIXBHIGH	MIXBH	1	4/3/01	09:33	
		MIXAHIGH	MIXAH	1	4/3/01	09:35	
		ICV	ICV	1	4/3/01	09:42	
		ICB	ICB	1	4/3/01	09:44	
		CRI1	CRI	1	4/3/01	09:54	
		ICSA1	ICSA	1	4/3/01	09:56	
		ICSAB1	ICSAB	1	4/3/01	09:59	
		CCV1	CCV	1	4/3/01	10:01	
		CCB1	CCB	1	4/3/01	10:04	
		IP010402-3	MB	1	4/3/01	10:09	IP010402-3
		IP010402-3	LCS	1	4/3/01	10:12	IP010402-3
		0103171-2	SMP	1	4/3/01	10:16	IP010402-3
		0103171-2	DUP	1	4/3/01	10:19	IP010402-3
		0103171-2	SER	5	4/3/01	10:22	IP010402-3
		0103171-2	MS	1	4/3/01	10:25	IP010402-3
		0103171-3	SMP	1	4/3/01	10:27	IP010402-3
		0103178-1	SMP	1	4/3/01	10:30	IP010402-3
		0103178-2	SMP	1	4/3/01	10:32	IP010402-3
		IP010402-1	MB	1	4/3/01	10:34	IP010402-1
		CCV2	CCV	1	4/3/01	10:37	
		CCB2	CCB	1	4/3/01	10:39	
		IP010402-1	LCS	1	4/3/01	10:41	IP010402-1
		0103178-3	SMP	1	4/3/01	10:44	IP010402-1
		0103178-3	DUP	1	4/3/01	10:46	IP010402-1
		0103178-3	SER	5	4/3/01	10:49	IP010402-1
		0103178-3	MS	1	4/3/01	10:51	IP010402-1
		0103178-4	SMP	1	4/3/01	10:53	IP010402-1
		0103178-5	SMP	1	4/3/01	10:56	IP010402-1
		CRI2	CRI	1	4/3/01	10:58	
		ICSA2	ICSA	1	4/3/01	11:00	
		ICSAB2	ICSAB	1	4/3/01	11:03	
		CCV3	CCV	1	4/3/01	11:05	
		CCB3	CCB	1	4/3/01	11:08	
		0103178-6	SMP	1	4/3/01	11:10	IP010402-1
		0103178-7	SMP	1	4/3/01	11:12	IP010402-1
		0103178-8	SMP	1	4/3/01	11:15	IP010402-1
		0103178-9	SMP	1	4/3/01	11:17	IP010402-1

Data Package ID: IT0103075-1

ICP Run Log -- 4/3/2001

Instrument ID: ICPTrace
 File Name: TS10403
 AnalRunID: IT010403-1A1
 CalibRefID: IT010403-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
		0103178-10	SMP	1	4/3/01	11:21	IP010402-1
		0103178-11	SMP	1	4/3/01	11:23	IP010402-1
		0103178-12	SMP	1	4/3/01	11:26	IP010402-1
		0103178-13	SMP	1	4/3/01	11:28	IP010402-1
		0103178-14	SMP	1	4/3/01	11:30	IP010402-1
		0103178-15	SMP	1	4/3/01	11:33	IP010402-1
		CCV4	CCV	1	4/3/01	11:35	
		CCB4	CCB	1	4/3/01	11:37	
		0103178-16	SMP	1	4/3/01	11:40	IP010402-1
		0103178-14	SMP	10	4/3/01	11:44	IP010402-1
		IP010402-2	MB	1	4/3/01	11:47	IP010402-2
		IP010402-2	LCS	1	4/3/01	11:49	IP010402-2
		0103179-1	SMP	1	4/3/01	11:51	IP010402-2
		0103179-2	SMP	1	4/3/01	11:54	IP010402-2
		0103179-3	SMP	1	4/3/01	11:56	IP010402-2
		CRI3	CRI	1	4/3/01	11:59	
		ICSA3	ICSA	1	4/3/01	12:01	
		ICSAB3	ICSAB	1	4/3/01	12:03	
		CCV5	CCV	1	4/3/01	12:06	
		CCB5	CCB	1	4/3/01	12:09	
		0103179-4	SMP	1	4/3/01	12:13	IP010402-2
		0103179-5	SMP	1	4/3/01	12:16	IP010402-2
		0103179-6	SMP	1	4/3/01	12:18	IP010402-2
		0103179-6	DUP	1	4/3/01	12:20	IP010402-2
		0103179-6	SER	5	4/3/01	12:29	IP010402-2
		0103179-6	MS	1	4/3/01	12:31	IP010402-2
		0103179-7	SMP	1	4/3/01	12:34	IP010402-2
		0103179-8	SMP	1	4/3/01	12:36	IP010402-2
		0103179-9	SMP	1	4/3/01	12:38	IP010402-2
		0103179-10	SMP	1	4/3/01	12:41	IP010402-2
		CCV6	CCV	1	4/3/01	12:43	
		CCB6	CCB	1	4/3/01	12:45	
		0103179-10	DUP	1	4/3/01	12:48	IP010402-2
		0103179-10	SER	5	4/3/01	12:50	IP010402-2
		0103179-10	MS	1	4/3/01	12:53	IP010402-2
		0103179-11	SMP	1	4/3/01	12:55	IP010402-2
		0103179-12	SMP	1	4/3/01	12:57	IP010402-2

Data Package ID: IT0103075-1

ICP Run Log -- 4/3/2001

Instrument ID: ICPTrace
 File Name: TS10403
 AnalRunID: IT010403-1A1
 CalibRefID: IT010403-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
		0103179-13	SMP	1	4/3/01	13:00	IP010402-2
		0103179-5	SMP	10	4/3/01	13:09	IP010402-2
		CRI4	CRI	1	4/3/01	13:12	
		ICSA4	ICSA	1	4/3/01	13:14	
		ICSAB4	ICSAB	1	4/3/01	13:17	
		CCV7	CCV	1	4/3/01	13:19	
		CCB7	CCB	1	4/3/01	13:21	
		0103179-12	SMP	50	4/3/01	13:25	IP010402-2
		0103179-6	A	1	4/3/01	13:28	IP010402-2
		0103179-10	A	1	4/3/01	13:30	IP010402-2
	D-03-S	0103075-8	SMP	10	4/3/01	13:33	IP010330-3
		CRI5	CRI	1	4/3/01	13:35	
		ICSA5	ICSA	1	4/3/01	13:37	
		ICSAB5	ICSAB	1	4/3/01	13:40	
		CCV8	CCV	1	4/3/01	13:42	
		CCB8	CCB	1	4/3/01	13:45	
		IP010403-2	MB	1	4/3/01	13:51	IP010403-2
		IP010403-2	LCS	1	4/3/01	13:53	IP010403-2
		0103197-1	SMP	1	4/3/01	13:56	IP010403-2
		0103197-2	SMP	1	4/3/01	13:58	IP010403-2
		0103197-3	SMP	1	4/3/01	14:00	IP010403-2
		0103197-4	SMP	1	4/3/01	14:03	IP010403-2
		0103156-1	SMP	1	4/3/01	14:05	IP010403-2
		0103156-2	SMP	1	4/3/01	14:08	IP010403-2
		0103156-3	SMP	1	4/3/01	14:10	IP010403-2
		0103156-4	SMP	1	4/3/01	14:12	IP010403-2
		CCV9	CCV	1	4/3/01	14:15	
		CCB9	CCB	1	4/3/01	14:17	
		0103156-5	SMP	1	4/3/01	14:20	IP010403-2
		0103156-7	SMP	1	4/3/01	14:22	IP010403-2
		0103156-8	SMP	1	4/3/01	14:24	IP010403-2
		0103194-1	SMP	1	4/3/01	14:27	IP010403-2
		0103194-2	SMP	1	4/3/01	14:29	IP010403-2
		0103194-3	SMP	1	4/3/01	14:31	IP010403-2
		0103194-4	SMP	1	4/3/01	14:34	IP010403-2
		0103194-5	SMP	1	4/3/01	14:36	IP010403-2
		0103194-5	DUP	1	4/3/01	14:39	IP010403-2

Data Package ID: IT0103075-1

ICP Run Log -- 4/3/2001

Instrument ID: ICPTrace

File Name: TS10403

AnalRunID: IT010403-1A1

CalibRefID: IT010403-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
		0103194-5	SER	5	4/3/01	14:41	IP010403-2
		CCV10	CCV	1	4/3/01	14:43	
		CCB10	CCB	1	4/3/01	14:46	
		0103194-5	MS	1	4/3/01	14:48	IP010403-2
		0103194-5	MSD	1	4/3/01	14:50	IP010403-2
		CCV11	CCV	1	4/3/01	14:53	
		CCB11	CCB	1	4/3/01	14:55	
		CCV12	CCV	1	4/3/01	15:33	
		CCB12	CCB	1	4/3/01	15:35	
		0103197-1	SMP	2	4/3/01	15:38	IP010403-2
		0103197-2	SMP	2	4/3/01	15:40	IP010403-2
		0103197-3	SMP	2	4/3/01	15:43	IP010403-2
		0103197-4	SMP	2	4/3/01	15:45	IP010403-2
		0103156-4	SMP	2	4/3/01	15:47	IP010403-2
		0103194-2	SMP	3	4/3/01	15:50	IP010403-2
		0103194-5	SMP	2	4/3/01	15:52	IP010403-2
		0103194-5	DUP	2	4/3/01	15:54	IP010403-2
		0103194-5	SER	10	4/3/01	15:57	IP010403-2
		0103194-5	MS	2	4/3/01	15:59	IP010403-2
		CCV13	CCV	1	4/3/01	16:02	
		CCB13	CCB	1	4/3/01	16:04	
		0103194-5	MSD	2	4/3/01	16:06	IP010403-2
		0103194-5	A	1	4/3/01	16:09	IP010403-2
		F010402-1	MB	1	4/3/01	16:13	IP010403-1
		F010402-1	LCS	1	4/3/01	16:16	IP010403-1
		0103167-1	SMP	1	4/3/01	16:18	IP010403-1
		0103167-1	DUP	1	4/3/01	16:20	IP010403-1
		0103167-1	SER	5	4/3/01	16:23	IP010403-1
		0103167-1	MS	1	4/3/01	16:25	IP010403-1
		0103167-1	MSD	1	4/3/01	16:27	IP010403-1
		0103167-2	SMP	1	4/3/01	16:30	IP010403-1
		CCV14	CCV	1	4/3/01	16:32	
		CCB14	CCB	1	4/3/01	16:35	
		0103167-3	SMP	1	4/3/01	16:37	IP010403-1
		0103167-4	SMP	1	4/3/01	16:39	IP010403-1
		0103167-3	SMP	2	4/3/01	16:45	IP010403-1
		0103167-4	SMP	2	4/3/01	16:47	IP010403-1

Data Package ID: IT0103075-1

ICP Run Log -- 4/3/2001

Instrument ID: ICPTTrace
File Name: TS10403
AnalRunID: IT010403-1A1
CalibRefID: IT010403-1A1

Comment	Field ID	Lab ID	QC Type	DF	Date Analyzed	Time Analyzed	Prep Batch ID
		CRI6	CRI	1	4/3/01	16:50	
		ICSA6	ICSA	1	4/3/01	16:52	
		ICSAB6	ICSAB	1	4/3/01	16:55	
		CCV15	CCV	1	4/3/01	16:57	
		CCB15	CCB	1	4/3/01	16:59	

Data Package ID: IT0103075-1

TARGET SHEET
EPA REGION VIII
SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 494266

SITE NAME: VASQUEZ BOULEVARD/INTERSTATE 70

DOCUMENT DATE: 08/16/2001

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☒ TYPES OF DOCUMENTS NOT TO BE SCANNED
(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

ANALYSIS REPORT



Paragon Analytics, Inc.

GC/MS Volatiles Case Narrative

Washington Group International, Inc.

EPA RAC -- 49941007

Order Number - 0103075

1. This report consists of 2 water samples and 6 sludge samples. The samples were received cool and intact by Paragon on 03/10/01.

All aqueous samples were free of head space prior to analysis.

2. These samples were prepared and analyzed according to SW-846, 3rd Edition procedures. Specifically, the water samples were prepared by purging 5 mls using purge and trap procedures based on Method 5030.

The sludge samples were extracted with methanol, which was then injected into the instrument using purge and trap procedures. The procedures for the extraction of soil and injection of the extract are based on Method 5030.

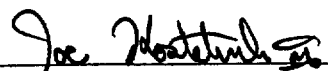
3. The samples were analyzed using GC/MS with a RTX-624 capillary column according to protocols based on SW-846 Method 8260B utilizing Paragon SOP 525 Rev 4. All positive results were quantitated with the average response of the initial calibration standards using the internal standard technique. The identification of positive results was achieved by a comparison of the retention time and mass spectrum of the sample versus the daily calibration standard.
4. All initial calibration criteria for SPCC's and CCC's were met. Method 8260B states that the average response factor may be used for quantitation for all analytes if the mean of the RSD values for all analytes is less than or equal to 15%. The initial calibration had a mean RSD value of less than 15%.
5. All continuing calibration criteria were met.

6. Methylene chloride, acetone and 2-butanone are common laboratory contaminants. In order to minimize the levels of these compounds detected in the gc/ms analysis, Paragon has designated its volatile laboratory as a restricted access area. In addition, the laboratory has been equipped with a dedicated, conditioned air intake and exhaust system that operates under positive pressure in order to minimize cross contamination of these compounds.

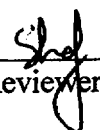
Method blank VL010319-1 and methanol blank VL010321-1M had methylene chloride detected below the reporting limit and method blank VL010321-1 had methylene chloride detected above the reporting limit. This compound was detected in the samples, so the data were flagged.

7. All laboratory control spike and laboratory control spike duplicate recoveries and RPDs were within the acceptance criteria.
8. All matrix spike and matrix spike duplicate recoveries and RPDs were within acceptance criteria.
9. The samples were analyzed within the established holding times.
10. All surrogate recoveries were within acceptance criteria.
11. All internal standard recoveries were within acceptance criteria.
12. Due to matrix interferences and the concentration of target analytes, samples 3, 4, 6, 7, 8, and 9 were analyzed at a higher dilution. The reporting limits have been adjusted accordingly.

The data contained in the following report have been reviewed and approved by the personnel listed below. In addition, Paragon Analytics, Inc. certifies that the analyses reported herein are true, complete and correct within the limits of the methods employed.


Joe Kostelnik
Organic Chemist

April 9, 2001
Date


Reviewer's Initials

4-6-01
Date

TARGET SHEET
EPA REGION VIII
SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 494266

SITE NAME: <u>VASQUEZ BOULEVARD/INTERSTATE 70</u>
DOCUMENT DATE: <u>08/16/2001</u>

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☒ TYPES OF DOCUMENTS NOT TO BE SCANNED
(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:



Paragon Analytics, Inc.

GC/MS Semivolatiles Case Narrative

Washington Group International, Inc.

EPA RAC -- 49941007

Order Number - 0103075

1. This report consists of 6 sludge samples. These samples were received cool and intact on 03/10/01.
2. These samples were prepared and analyzed according to SW-846, 3rd Edition protocol utilizing Paragon Standard Operating Procedures. Specifically, the sludge samples were diluted with solvent based on Method 3580. These extracts were then processed using GPC cleanup by Method 3640 in an attempt to remove potential interferences.
3. The extracts were analyzed using GC/MS with a DB-5.625 capillary column according to Paragon Standard Operating Procedure 506 Revision 8 based on SW-846 Method 8270C. All positive results were quantitated against the initial calibration standards using the internal standard technique. The identification of positive results was achieved by a comparison of the retention time and mass spectrum of the sample versus the daily calibration standard.
4. All initial calibration criteria were met. Method 8270C states that if the average of the percent relative standard deviations (RSDs) is less than 15, the average response factors may be used for quantitation. We quantitated these compounds using the average responses.
5. All continuing calibration criteria were met.
6. There were no target compounds detected in the method blank.
7. All laboratory control spike and laboratory control spike duplicate recoveries and RPDs were within the acceptance criteria.

8. All matrix spike and matrix spike duplicate recoveries and RPDs were within acceptance criteria with the following exceptions:

Spiked Compound	QC Sample	Direction
4-Nitrophenol	8MS/MSD	Low

The recoveries of these compounds in the laboratory control spike and laboratory control spike duplicate were within control limits, which suggests the outliers in the matrix spikes were due to matrix effects. No further action was taken. Laboratory control spike and laboratory control spike duplicate results have been included.

9. The samples were extracted and analyzed within the established holding times.
10. All surrogate recoveries were within acceptance limits with the following exceptions:

Surrogate	Sample	Direction
2-Fluorophenol	8MSD	Low
2,4,6-Tribromophenol	3, 4, 8MS/MSD	Low

The surrogate 2,4,6-tribromophenol in sample 8 was within the acceptance criteria but trended low, which suggests matrix effects are present in the sample. Re-extraction was not required.

The re-analysis of the samples 3 and 4 confirmed the original surrogate analysis.

11. All internal standard recoveries were within acceptance criteria.

The data contained in the following report have been reviewed and approved by the personnel listed below. In addition, Paragon Analytics, Inc. certifies that the analyses reported herein are true, complete and correct within the limits of the methods employed.

Gayle Cheng
Gayle Cheng
Organic Chemist

4-9-2001
Date

Sh
Reviewer's Initials

4-5-01
Date

TARGET SHEET
EPA REGION VIII
SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 494266

SITE NAME: VASQUEZ BOULEVARD/INTERSTATE 70

DOCUMENT DATE: 08/16/2001

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☒ TYPES OF DOCUMENTS NOT TO BE SCANNED
(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:



Paragon Analytics, Inc.

Pesticides Case Narrative

Washington Group International, Inc.

EPA RAC -- 49941007

Order Number - 0103075

1. This report consists of 2 liquid waste samples and 2 solid waste samples. The samples were received cool and intact by Paragon on 03/10/2001.
2. These samples were extracted and analyzed according to SW-846, 3rd Edition procedures. Specifically, the liquid waste samples were diluted with solvent based on Method 3580. The solid waste samples were extracted using soxhlet procedures according to Paragon Analytics, Inc. Standard Operating Procedure 625 Revision 4 based on Method 3540C.

The extracts were then processed using florisil cleanup following Paragon Analytics, Inc. Standard Operating Procedure 648 Revision 2 based on Method 3620B in an attempt to remove potential interferences.

3. The extracts were then analyzed using GC/ECD (electron capture detectors) with a RTX-CLPesticides capillary column according to Paragon Analytics, Inc. Standard Operations Procedure 402 Revision 5 based on Method 8081A. All positive results were then confirmed on a RTX-CLPesticides II column. The quantitation of each analyte is the lower of the concentrations obtained from each column which met initial and continuing calibration criteria. This minimizes the chances of reporting elevated results based on interferences.
4. The breakdown for endrin and 4,4'-DDT met acceptance criteria.
5. All initial and continuing calibration criteria were met with the following exceptions:

Continuing calibration 032601-2CCV - methoxychlor was out high on column 1.
Continuing calibration 032601-3CCV - methoxychlor was out high on column 1.
Continuing calibration 032601-4CCV - methoxychlor was out high on column 1.
Continuing calibration 032901-1CCV - methoxychlor was out high on column 1.

Continuing calibration 032901-2CCV - endosulfan sulfate was out high on column 1.
 Continuing calibration 033001-1CCV - 4,4'DDD was out low on column 2.
 Methoxychlor and endosulfan sulfate were out high on column 1.
 Continuing calibration 033001-2CCV - aldrin, heptachlor epoxide, gamma chlordane, alpha chlordane, 4,4'DDE, dieldrin, endrin, 4,4'DDD, endosulfan II, and endrin ketone were out low on column 2.
 Quantitation for each analyte was reported from the column that passed initial and continuing calibration criteria.

Continuing calibration 033001-2CCV - endosulfan I, endrin aldehyde, and decachlorobiphenyl were out low on both columns.

Samples 1, 2, and matrix spikes were bracketed by the above calibration verification. The samples were analyzed on a separate day with similar results in the ending calibration verification. The raw data for the sequence and ending calibration verification are included in the miscellaneous section of this report.

6. The method blanks associated with this project were below the reporting limits for all analytes.
7. All laboratory control spike and laboratory control spike duplicate recoveries and RPDs were within the acceptance criteria.
8. A matrix spike duplicate could not be performed on the waste liquid samples because of insufficient sample. A matrix spike, laboratory control spike, and laboratory control spike duplicate were performed instead.

All matrix spike and matrix spike duplicate recoveries and RPDs were within acceptance criteria with the following exceptions:

Spiked Compound	QC Sample	Direction
gamma-BHC	0103075-11MS	low
dieldrin	0103075-11MS	low
dieldrin	0103075-1MS & 0103075-1MSD	low

The recoveries of these compounds in the laboratory control spike and laboratory control spike duplicate were within control limits, which suggest the outliers in the matrix spikes may have been due to matrix effects. No further action was warranted. Blank spike and blank spike duplicate results have been included.

Spiked Compound	QC Sample	Direction
gamma-BHC	0103075-1MS & 0103075-1MSD	RPD

Gamma-BHC was within the control limits in each of the matrix spikes. As no sample quantitations are compromised and reporting limits are defensible, data are submitted.

9. All samples were extracted and analyzed within the established holding times.
10. Surrogate recoveries could not be reported for sample 2 due to sample dilutions.

All surrogate recoveries were within acceptable limits with the following exception:

Surrogate	Sample	Direction
decachlorobiphenyl	0103075-1	high

The method states that one surrogate may be outside control limits without further action.

11. Samples 2 and 12 were analyzed at a higher dilution in order to bring target analytes within the calibration range of the instrument. The reporting limits have been adjusted accordingly.

The data contained in the following report have been reviewed and approved by the personnel listed below. In addition, Paragon Analytics, Inc. certifies that the analyses reported herein are true, complete and correct within the limits of the methods employed.

for P. Sheneman
Dan Sheneman
GC Analyst

10 Apr 2001
Date

EX
Reviewer's Initials

0409-01
Date

TARGET SHEET
EPA REGION VIII
SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 494266

SITE NAME: <u>VASQUEZ BOULEVARD/INTERSTATE 70</u>
DOCUMENT DATE: <u>08/16/2001</u>

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- ☐ PHOTOGRAPHS
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(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:



Paragon Analytics, Inc.

PCBs Case Narrative

Washington Group International, Inc.

EPA RAC -- 49941007

Order Number - 0103075

1. This report consists of 2 liquid waste samples and 2 solid waste samples. The samples were received cool and intact by Paragon on 03/10/2001.
2. These samples were extracted and analyzed according to SW-846, 3rd Edition procedures. Specifically, the liquid waste samples were diluted with solvent based on Method 3580. The solid waste samples were extracted using soxhlet procedures according to Paragon Analytics, Inc. Standard Operating Procedure 625 Revision 4 based on Method 3540C.

The extracts were then processed using sulfuric acid cleanup according to Paragon Analytics Standard Operating Procedure 651 Revision 4 based on Method 3665 in an attempt to remove potential interferences.

3. The extracts were then analyzed using GC/ECD (electron capture detectors) with a RTX-CLPesticides capillary column according to Paragon Analytics Standard Operating Protocol 409 Revision 0 based on SW-846 Method 8082. All positive results were then confirmed on a RTX-CLPesticidesII column. The quantitation of each analyte is the lower of the concentrations obtained from each column which met initial and continuing calibration criteria. This minimizes the chances of reporting elevated results based on interferences.

4. All initial and continuing calibration criteria were met with the following exceptions:

Continuing calibration 1254 040201-2CCV - aroclor 1254 was out high on both columns.

Because the sensitivity of the instrument increased and no target compounds were detected, no further action was taken. Reporting limits are supported.

Continuing calibration 1254 040201-5CCV - aroclor 1254 was out low on both columns.

Continuing calibration 1660 040201-5CCV - aroclor 1016 was out low on column 2.

Aroclor 1260 and decachlorobiphenyl were out low on both columns.

All samples and matrix spikes were bracketed by the above calibration verification. The samples were analyzed on a separate day with similar results in the ending calibration verification. The raw data for the sequence and ending calibration verification are included in the miscellaneous section of this report.

5. The method blanks associated with this project were below the reporting limits for all analytes.
6. All laboratory control spike and laboratory control spike duplicate recoveries and RPDs were within the acceptance criteria.
7. Matrix spikes and matrix spike duplicates could not be performed on the waste liquid samples because of insufficient sample. A laboratory control spike and laboratory control spike duplicate were performed instead.

All solid waste matrix spike and matrix spike duplicate recoveries and RPDs were within acceptance criteria with the following exception:

Spiked Compound	QC Sample	Direction
aroclor 1260	0103075-1MSD	low

The recoveries of this compound in the laboratory control spike and laboratory control spike duplicate were within control limits, which suggest the outlier in the matrix spike duplicate may have been due to matrix effects. No further action was warranted. Blank spike and blank spike duplicate results have been included.

8. All samples were extracted and analyzed within the established holding times.
9. All surrogate recoveries were within acceptable limits with the following exception:

Surrogate	Sample	Direction
decachlorobiphenyl	0103075-1	high

The method states that one surrogate may be outside control limits without further action.

The data contained in the following report have been reviewed and approved by the personnel listed below. In addition, Paragon Analytics, Inc. certifies that the analyses reported herein are true, complete and correct within the limits of the methods employed.

Dan Sheneman

Dan Sheneman
GC Analyst

4-9-01

Date

EX
Reviewer's Initials

04-09-01
Date

TARGET SHEET
EPA REGION VIII
SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 494266

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(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

ENCLOSURE 5:

State of Utah Certification



State of Utah

DIVISION OF EPIDEMIOLOGY
AND LABORATORY SERVICES

June 1, 2001

Michael O. Leavitt
Governor

Rod L. Betit
Executive Director

Charles D. Brokopp, Dr. P.H.
Director

Bureau of Laboratory Improvement
46 North Medical Drive
Salt Lake City, Utah 84113-1105
Telephone: (801) 584-8469
Fax: (801) 584-8501

Paragon Analytics Incorporated
Donald F Gipple Director
225 Commerce Drive
Fort Collins CO 80524

ID # ATL2
Account # 3034901511

Director,

On the basis of your most recent audit results and compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Safe Drinking Water Act and authorized to perform the following analytes, or groups of analytes by method:

Radionuclides

900.0

Gross Alpha & Beta

901.1

Cesium 134

Gamma Emitters

906.0

Tritium

D-3972-90

Uranium

This laboratory's certification date is effective: 05/31/2001.

The analytes or groups of analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. Any discrepancies must be documented and notice received by this Bureau within 15 days of receipt. The certification will be recalled in the event your laboratory's certification is revoked.

Respectfully,

Charles Brokopp, Dr. P.H.

The expiration for the laboratory's certification is 10/31/2001. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data user to verify the most current certification letter for the authorized method. Please call 801-584-8469.



State of Utah

DIVISION OF EPIDEMIOLOGY
AND LABORATORY SERVICES

June 1, 2001

Michael O. Leavitt
Governor

Rod L. Betit
Executive Director

Charles D. Brokopp, Dr. P.H.
Director

Bureau of Laboratory Improvement
46 North Medical Drive
Salt Lake City, Utah 84113-1105
Telephone: (801) 584-8485
Fax: (801) 584-8501

Paragon Analytics Incorporated
Donald F Gipple Director
225 Commerce Drive
Fort Collins CO 80524

ID # ATL2
Account # 3034901511

Director,

On the basis of your most recent audit results and compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Clean Water Act and authorized to perform the following analytes, or groups of analytes by method:

Radiological

Method 903.0

Total Radium

This laboratory's certification date is effective: 05/31/2001.

The analytes or groups of analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. Any discrepancies must be documented and notice received by this Bureau within 15 days of receipt. The certification will be recalled in the event your laboratory's certification is revoked.

Respectfully,


Charles Brokopp, Dr. P.H.



State of Utah

DIVISION OF EPIDEMIOLOGY
AND LABORATORY SERVICES

June 1, 2001

Michael O. Leavitt
Governor

Rod L. Betit
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Fax: (801) 584-8501

Paragon Analytics Incorporated
Donald F Gipple Director
225 Commerce Drive
Fort Collins CO 80524

ID # ATL2
Account # 3034901511

Director,

On the basis of your most recent audit results and compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Resource Conservation and Recovery Act and authorized to perform the following analytes, or groups of analytes by method:

Characteristics

1010

Pensky-Martens Closed Cup Method for
Determining Ignitability

1311

Toxicity Characteristic Leaching
Procedure Metals
Toxicity Characteristic Leaching
Procedure Semi-Volatiles
Toxicity Characteristic Leaching
Procedure Volatiles

Sec 8.3

Reactivity

Inorganic

9010 B

Total and Amenable Cyanide: Distillation

9013

Cyanide Extraction Procedure for Solids
and Oils

9020 B

Total Organic Halides (TOX)

9040 B

pH Electrometric Measurement

9045 C

Soil and Waste pH

9050 A

Specific Conductance

9056

Determination of Inorganic Anions by IC
(Bromide)

Determination of Inorganic Anions by IC
(Chloride)

Determination of Inorganic Anions by IC
(Fluoride)

Determination of Inorganic Anions by IC
(Nitrate)

Determination of Inorganic Anions by IC
(Nitrite)

Determination of Inorganic Anions by IC
(Phosphate)

Determination of Inorganic Anions by IC
(Sulfates)

9071 A

Oil and Grease Extraction Method for
Sludge and Sediment Samples

9095 A

Paint Filter Liquids Test

Metal Digestion

3005 A

Acid Digestion Total Recoverable or
Dissolved Metals

3010 A

Acid Digestion for Total Metals

3020 A

Acid Digestion for Total Metals

3050 B

Acid Digestion of Sediments, Sludges
and Soils

3060 A

Alkaline Digestion for Hexavalent
Chromium

The expiration for the laboratory's certification is 10/31/2001. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data user to verify the most current certification letter for the authorized method. Please call 801-584-8469.

Metals

6010 B

Aluminum
Antimony
Arsenic
Barium
Beryllium
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Molybdenum
Nickel
Potassium
Selenium
Silver
Sodium
Strontium
Thallium
Vanadium
Zinc

6020

Manganese

7060 A

Arsenic

7196 A

Chromium Hexavalent Colorimetric

7421

Lead

7470 A

Mercury

7471 A

Mercury

7740

Selenium

7841

Thallium

Organic Cleanup

3620 B

Florisil Cleanup

3630 C

Silica Gel Cleanup

3640 A

Gel Permeation Cleanup

3650 B

Acid Base Partition Cleanup

3660 B

Sulfur Cleanup

Organic Extraction

3510 C

Separatory Funnel Liquid-Liquid
Extractions

3520 C

Continuous Liquid-Liquid Extraction

3540 C

Soxhlet Extraction

3550 B

Ultrasonic Extraction

Organic Instrumentation

8015 B

Nonhalogenated Organics Using GC/FID

8021 B

Aromatic and Halogenated Volatiles by
GC using Photoionization and or ECD:

8081 A

Organochlorine Pesticides By Capillary
Column Gas Chromatography

8082

PCBs By Capillary Column Gas
Chromatography

8141 A

Organophosphorus Compounds By GC:
Capillary Column Technique

8151 A

Chlorinated Herbicides By GC Using
Methylation Or Pentafluorobenzoylation

8260 B

Volatile Organic Compounds by GC/MS:
Capillary Column Technique

8270 C

Semivolatile Organic Compounds By Gas
Chromatography/Mass Spectrometry

8310

Polynuclear Aromatic Hydrocarbons

8330

Explosives

Radiochemistry

9310

Gross Alpha and Gross Beta

9315

Alpha Emit Radium Isotope

Volatile Organic Preparation

5030 B

Purge-and-Trap for Aqueous Samples

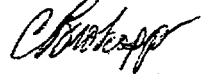
5035

Purge-and-Trap and Extraction for
Volatile Organics in Soil & Waste

This laboratory's certification date is effective: 05/31/2001.

The analytes or groups of analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. Any discrepancies must be documented and notice received by this Bureau within 15 days of receipt. The certification will be recalled in the event your laboratory's certification is revoked.

Respectfully,



Charles Brokopp, Dr. P.H.

ENCLOSURE 6:

Paragon SOP 409 and SOP 525

PARAGON ANALYTICS, INC.
STANDARD OPERATING PROCEDURE 409 REVISION 0

TITLE: ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBs)
BY GAS CHROMATOGRAPHY -- METHOD 8082

FORMS: NONE

APPROVED BY: TECHNICAL MANAGER *Debra Schmitt* DATE 2-15-99
QUALITY ASSURANCE MANAGER _____ DATE _____
LABORATORY MANAGER _____ DATE _____

HISTORY: Rev 0, 02/15/99.

1.0 SCOPE AND APPLICATION

- 1.1 This standard operating procedure (SOP) and the method it references -- Method 8082 -- are used to determine the concentration of Aroclors 1016 through 1260 in liquid and solid matrices.
- 1.2 The following selected compounds may be analyzed by this method:
- | | |
|--------------|--------------|
| Aroclor 1016 | Aroclor 1248 |
| Aroclor 1221 | Aroclor 1254 |
| Aroclor 1232 | Aroclor 1260 |
| Aroclor 1242 | |

2.0 SUMMARY OF METHOD

2.1 OVERVIEW

Extracted samples that have been concentrated are directly injected into a gas chromatograph (GC) containing a splitter and two columns. Each column separates the target analytes that are then detected by an electron capture detector (ECD). This dual column chromatography allows tentative identification (by first column) and confirmation (by second column) to be performed simultaneously.

2.2 SAMPLE PREPARATION

- 2.2.1 Liquid Samples: One (1) liter of sample is extracted at neutral pH with methylene chloride using a continuous liquid extractor or a separatory funnel. The extract is concentrated and solvent exchanged into hexane for analysis.
- 2.2.2 Solid Samples: A 2-30 g aliquot of homogenized sample is extracted with methylene chloride using pulse sonication or soxhlet extractor. The extract is concentrated and solvent exchanged into hexane for analysis.

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Note: Usually, a 30 g aliquot is used for soil samples; a 2 g aliquot is used for paint chips.

3.0 RESPONSIBILITIES

- 3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.
- 3.2 Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this method. This demonstration may come in the form of supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency evaluation test.
- 3.3 Final review and sign-off of the data are performed by the department supervisor or designee. Initialing and dating the file indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.
- 3.4 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4.0 INTERFERENCES

- 4.1 Interferences from phthalate esters can be minimized by using plastic-free solvent containers and scrupulously cleaned glassware that has been solvent rinsed prior to use.
- 4.2 Sulfuric acid clean up techniques may be used to remove interferences caused by the presence of organochlorine and/or organophosphorous pesticides.
- 4.3 Elemental sulfur (particularly in sediment samples) may interfere and can be removed by using appropriate clean up techniques prior to sample analysis.

5.0 APPARATUS AND MATERIALS

- 5.1 GAS CHROMATOGRAPH/DETECTORS
Hewlett Packard 5890 Series II GC or equivalent equipped with dual on-column injection and electron capture detectors (ECDs).
- 5.2 ELECTRONIC INTEGRATOR
Any data acquisition system capable of acquiring, storing and processing

chromatographic data (e.g., Hewlett Packard Chem Station or equivalent).

5.3 CAPILLARY COLUMN

Primary: RTx-35 or equivalent (i.e., 30 m, 0.53 mm ID, 0.5 μ m)

Confirmation: RTx-5 or equivalent (i.e., 30 m, 0.53 mm ID, 1.5 μ m)

5.4 GASES

Helium (ultra high purity; used as carrier gas)

Nitrogen (ultra high purity; used as make-up gas)

5.5 AUTOMATED SAMPLER

Hewlett Packard 7673 Automated Injection System or equivalent.

5.6 MEASURING DEVICES

5.6.1 Precision Hamilton (or equivalent) microsyringes in 1 μ L, 5 μ L and 1.0 mL sizes.

5.6.2 Volumetric flasks, Class A with ground glass stoppers, 10 mL and 25 mL sizes.

6.0 REAGENTS

6.1 SOLVENTS

Methylene Chloride

n-Hexane

Isooctane

Methanol

Note: Only pesticide grade solvents may be used.

6.2 STOCK AND INTERMEDIATE STANDARDS

6.2.1 Prepared from EPA repository standards or certified vendor solutions. Stored in PFTE (Teflon)-sealed vials in the dark at 4 °C. Undiluted stock standards may be retained for up to one year; diluted standards for up to 6 months. Standards may need replaced sooner if laboratory quality control sample analyses indicate deterioration.

6.2.2 Stock Standards: An approximately 1000 mg/L (per component) stock solution is purchased from a suitable vendor or prepared in-house gravimetrically by accurately weighing 0.0100 g of pure material into a 10 mL Class A volumetric flask and diluting to volume with n-hexane or isooctane. If purity of the compound is 96% or greater, no weight correction is necessary; if compound purity is less than 96%, concentration must be corrected mathematically based on weight used.

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A combination standard containing Aroclor 1016 and 1260 will generate peaks covering the range of all Aroclors of interest. Individual standards for all Aroclors may be created, however, to assist in pattern recognition. The stock standards are subsequently diluted to create the intermediate stock standards.

- 6.2.3 Intermediate Stock Standards: Generally prepared by diluting 1 mL of stock standard to 25 mL using a Class A volumetric flask and n-hexane or isooctane. The intermediate stock standard is further diluted to create the calibration standards.

6.3 CALIBRATION STANDARDS

- 6.3.1 Calibration Standards: Prepared at a minimum of 5 different concentrations bracketing the linear range of the detector. The lowest concentration standard shall be at a level at or below the analyte reporting limit. Create calibration standards by diluting aliquots of the intermediate stock standard to volume using a Class A volumetric flask and n-hexane or isooctane. A calibration standard at a concentration level midpoint of the calibration curve will be used as a continuing calibration verification (CCV) standard.

- 6.3.2 Independent Calibration Verification Standard (ICV): Certified and purchased from a vendor or made gravimetrically in-house. Uses a source different from that of the calibration standard so that the accuracy of the calibration standard may be independently verified. Created and analyzed at a concentration level that is the midpoint of the calibration range.

6.4 SURROGATE SPIKE STANDARD

Surrogate Spike Standard: Certified and purchased from a vendor or made in-house. Contains 500 ng/ μ L each tetrachloro-m-xylene and decachlorobiphenyl in methanol. During preparation, 1.0 mL of this standard is spiked into each sample, standard and quality control sample.

Note: An internal standard is not used for Aroclor analysis. Aroclor content is determined by pattern recognition and quantitation is accomplished using the external standard method.

7.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

- 7.1 Samples must be collected according to an approved sampling plan.
- 7.2 Liquid samples are not chemically preserved and must be collected in amber glass containers (generally 1 L) with Teflon-lined lids. Samples must be maintained at

4 °C and extracted within 7 days of collection. Extracts must also be maintained at 4 °C and analyzed within 40 days of preparation.

- 7.3 Solid samples are collected in 250 mL widemouth glass containers with Teflon-lined lids. Solid samples are not chemically preserved and must be maintained at 4 °C. Solid samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

8.0 PROCEDURE

8.1 GAS CHROMATOGRAPHIC CONDITIONS

Carrier Gas (He):	1 - 6 mL/min.
Make-up Gas (N ₂):	20 - 40 mL/min.
Injector Temperature:	220 °C
Oven Temperature Program	
Initial Temperature:	110 °C
Oven Ramp:	20 °C/min. to 150 °C
Oven Ramp A:	7 °C/min. to 220 °C
Oven Ramp B:	5 °C/min. to 270 °C
Hold:	4 min.
Detector Temperature:	310 °C

8.2 INITIAL CALIBRATION

Prepare calibration standards as discussed above (including addition of surrogate). Inject 1 - 2 µL directly into the GC and analyze. Quantitation is accomplished via the external standard method of quantitation. Analyte calibration factors (CFs) are calculated as follows:

$$CF = \frac{\text{Sum of Selected Peak Areas or Heights}}{\text{Mass of Aroclor Injected On-Column (ng)}}$$

If the CFs over the working range of the detector are constant (i.e., $\leq 20\%$ RSD), then response is assumed to be invariant and the average (mean) CF may be used to quantitate sample content. Relative Standard Deviation (RSD) is calculated as:

$$RSD (\%) = \frac{\text{Standard Deviation (SD)}}{\text{Average (mean) CF}} \times 100$$

When RSD over the calibration range is greater than 20%, linearity through the origin cannot be assumed. It is then necessary to calculate analyte linearity

using a regression equation that does **not** pass through the origin (e.g., the least squares method). The regression calculation will yield a correlation coefficient (r) that must be ≥ 0.99 to be used for sample quantitation. Note that the correlation coefficient is an expression of "goodness of fit" with perfect fit being a value of 1.0.

8.3 INITIAL CALIBRATION VERIFICATION (ICV)

An ICV is run immediately after multi-point calibration. To be valid, the response of the second source ICV standard cannot differ from that of the midpoint of the first source initial calibration standard by more than 15%. The equation below is used to calculate Percent Difference (%D):

$$\%D = \frac{|(\text{ICV Response}) - (\text{Initial Calibration Response})|}{\text{Initial Calibration Response}} \times 100$$

If the % D of the ICV is $> 15\%$, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated.

8.4 CONTINUING CALIBRATION VERIFICATION (CCV)

The CCV monitors detector response during a run sequence. The concentration of this standard is at the midpoint of the initial calibration. After an acceptable ICV is analyzed, up to 10 samples may be analyzed. After the 10th sample, a CCV must be analyzed and the percent difference calculated. If the %D for the CCV is acceptable (i.e., $\pm 15\%$), another 10 field samples may be analyzed followed by the analysis of another CCV to bracket the sample analyses.

If any CCV does not meet acceptance criteria, analyses must be halted and the source of the problem found and corrected. The instrument must be recalibrated, and all samples injected since the last acceptable CCV must be reanalyzed.

8.5 RETENTION TIME WINDOWS

Retention Time Windows (RTWs) are established by analyzing a mid-level standard for each Aroclor, non-consecutively, over a 72 hour period. The standard deviation of these analyses is calculated based on the absolute retention time of selected peaks yielded for the Aroclor. Each Aroclor's RTW is defined as three times the calculated standard deviation.

8.6 SURROGATE RECOVERY

All control sample recoveries must be within established control limits. If the surrogate percent recovery is outside limits, the sample is reanalyzed to determine analytical error or matrix effect, the data is flagged as such and a

notation is made in the narrative comments. Percent Recovery (%R) is calculated as follows:

$$\%R = \frac{\text{Found Analyte Concentration}}{\text{Target (Anticipated) Analyte Concentration}} \times 100$$

8.8 CALCULATIONS AND REPORTING

8.8.1 Aroclors are identified through pattern recognition. Tentative identification occurs when selected peaks from a concentrated sample extract fall within the RTW of one column. If selected peak retention time also falls within their RTW on the second column (and the concentration is within a two fold window), the analyte's presence has been confirmed. Quantitation is calculated from both column responses and the value being impacted by the least amount of interference is reported. For the multi-response Aroclors, three to eight peaks are used for identification/quantitation. The same selected peaks must be consistently used for quantitation between the standard and sample set.

Note: Analyst expertise is crucial in identifying and quantitating samples containing multiple Aroclors or Aroclors that are particularly weathered.

8.8.2 SAMPLE ANALYSIS

Generally, 1 - 2 μL of the concentrated sample extract is directly injected into the GC via the automated injector. Where necessary, dilute sample extracts to keep response within the linear range. All prepared extracts contain the surrogate.

Sample concentration is calculated using the following equation:

$$\text{Concentration}_{\mu\text{g/L}} = \frac{[(A_x)(V_t)(DF)]}{(\text{mean CF})(V_s \text{ or } W_s)}$$

$\mu\text{g/Kg}$

Where:

A_x	=	analyte response (area units or peak height)
V_t	=	volume of total concentrated extract (μL)
DF	=	Dilution Factor (if applicable); if no dilution was made, DF = 1 (dimensionless)
mean CF	=	average standard response (area units or peak height)
V_s or W_s	=	(volume or weight) of sample extracted (mL or g)

9.0 QUALITY CONTROL

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9.1 DEFINITION OF ANALYSIS BATCH

For this method, an analysis batch is defined as a group of 20 or less field samples that are associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS) and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps.

9.2 DEFINITION OF ANALYTICAL SEQUENCE

The sequence of certain analyses is defined in the method. To be compliant, field, calibration and quality control samples shall be analyzed in the following repetitive sequence:

Reagent Blank
ICV
CCV
MB
LCS
(up to 10) Samples
CCV
(up to 10) Samples
MS*
Duplicate*
CCV

- * One MS and Duplicate analysis must be performed per batch of twenty samples or less of like matrix. These two quality control analyses may be performed at any time in the analytical sequence following daily calibration.

9.3 BLANKS

Method blanks are aliquots of matrix (i.e., organic-free water for liquids analyses; Ottawa Sand for solids analyses), which have been prepared and analyzed in the same manner as the associated field samples. MBs are run before processing any samples to demonstrate that interferences are under control. Each time a batch of samples is analyzed, extracted, or there is a change in reagents, an MB should be analyzed.

To be acceptable, concentrations of analytes of interest detected (if any) in the MB must be below the analyte reporting limit. If this criteria is not met, analyses must be halted and the source of the contamination found and corrected.

Two other blank types bear mention. Reagent blanks are simply an injection of solvent analyzed to show that the analytical system is free from contamination.

Carryover blanks are simply aliquots of contaminant-free matrix (which are not surrogate spiked) that are analyzed to clean the analytical system. These blanks are run as necessary and neither is evaluated against reporting limit criteria.

9.4 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) is analyzed to measure the accuracy of the method. The LCS is similar to the matrix spike analysis in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated.

9.5 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this analysis, either a field sample containing target compound contamination may be analyzed in duplicate, or the laboratory control sample or matrix spike analysis can be performed in duplicate. Relative Percent Difference (RPD) of the duplicate pair is calculated as follows:

$$\text{RPD (\%)} = \frac{|\text{Concentration}_x - \text{Concentration}_{\text{dup}}|}{(\text{Concentration}_x + \text{Concentration}_{\text{dup}}) / 2}$$

Where:

Concentration_x = analyte concentration in sample
Concentration_{dup} = analyte concentration in duplicate

9.6 MATRIX SPIKE

Matrix spikes consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection. One MS is analyzed per batch. Percent Recovery (%R) for spiked analytes is calculated as follows:

$$\%R = \frac{A_{\text{found}} - A_{\text{sample}}}{A_{\text{target}}} \times 100$$

Where:

A_{found} = Calculated analyte concentration in the MS or MSD sample
A_{sample} = Calculated analyte concentration in the unspiked field sample
A_{target} = The target (anticipated) concentration of the added analyte spike

Advisory acceptance criteria for all spikes and duplicates must be met. If MS

recovery or relative percent difference criteria are not met, results of the laboratory control sample analyses must be carefully considered. If LCS results are acceptable, a sample matrix interference is suspected and a notation in the narrative comments is made.

Note: In the event that not enough sample volume is provided to generate MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the narrative.

Also note that for projects in which the client is to designate MS/MSD samples, an analysis batch may not contain an MS/MSD pair. Where this occurs, a notation will be made in the narrative.

- 9.7 A method detection limit (MDL) study shall consist of the analysis of a blank and a minimum of seven replicate analyses for a target analyte at a concentration level near the capabilities of the method. The MDL study should be performed as needed and at a minimum, annually.

10.0 DEVIATIONS FROM METHOD

- 10.1 This SOP meets the requirements of Method 8082. There are no known deviations from the method.

11.0 SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

- 11.1.1 Read the MSDSs before prior to preparing standards or using any solvents or reagents for the first time.
- 11.1.2 Wear gloves, safety glasses, and lab coat when working with any chemical materials (e.g., standards, solvents, reagents, or samples), handling materials or equipment potentially contaminated with chemicals or within a laboratory area.
- 11.1.3 Any chemicals with a Threshold Limit Value (TLV) of less than 50 ppm shall be worked with in a laboratory fume hood (e.g., solvents and acids). All flammable compounds must be kept away from ignition sources.
- 11.1.4 Any non original containers used to hold reagents (e.g., wash bottles or automatic dispenser bottles) shall be labeled at a minimum with compound name, NFPA Health, Flammability and Reactivity ratings, and date.

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- 11.1.5 All compressed gas cylinders must be secured at all times a regulator is in place. The cylinder cap must be installed immediately after removing the regulator and before removing the tie down strap or chain from the cylinder. The cylinder shall be secured to a gas cart for transport. The cylinder must be stored capped and secured at all times.

11.2 WASTE DISPOSAL

- 11.2.1 Any hexane or other nonhalogenated organic solvents that has not been potentially contaminated with PCBs may be disposed of in the Acetonitrile/Nonhalogenated Waste. (Profile #AJ6738).
- 11.2.2 The extract vials and associated extracts that do not contain PCBs greater than 50 ppm may be disposed of intact in the Discarded Extract Vial Waste. (Profile # AJ6739).
- 11.2.3 The extract vials, associated extracts, and any PCB contaminated debris that may contain PCBs in excess of 50 ppm shall be disposed of intact in the PCB Debris Waste. (Profile # BS5030).
- 11.2.4 All empty solvent bottles are disposed of according to the appropriate SOPs. Please note that all labels and markings must be defaced prior to disposal.

12.0 REFERENCES

- 12.1 US EPA SW-846, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods", 3rd edition, Final Update III, Method 8082, Revision 0, December 1996.

Analytical Method: SW8082	Parameter: Polychlorinated Biphenyls (PCBs)	Summary of Internal Quality Control (QC) Procedures and Corrective Actions	
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration; minimum 5-point; all analytes	As needed (i.e., when daily calibration does not meet criteria)	a) $RSD \leq 20\%$, use mean CFs to quantitate. b) If $RSD > 20\%$ calculate linear regression (not forced through origin); use for quantitation if correlation coefficient (r) is ≥ 0.99 .	Evaluate/correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Initial Calibration Verification (ICV); run at midpoint of calibration	Daily prior to sample analyses	If $\pm 15\%$ D analyses may proceed.	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Continuing Calibration Verification (CCV); run at midpoint of calibration	Brackets each set of 10 field sample analyses	If $\pm 15\%$ D analyses may proceed.	Evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard column of the GC, prepare a new standard) and reanalyze. If CCV still non-compliant, recalibrate using a new curve. Samples analyzed after a failed CCV will be reanalyzed. If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported; and samples between the failed CCV and subsequent compliant CCV will be

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Analytical Method: SW8082	Parameter: Polychlorinated Biphenyls (PCBs)	Summary of Internal Quality Control (QC) Procedures and Corrective Actions	
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
			<p>reanalyzed.</p> <p>If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.</p>
Retention Time Window (RTW)	Whenever a new column is installed, based on 3 injections throughout a 72-hour period to be more representative of daily operations	<p>Column and compound specific. Window is $\pm 3x$ the standard deviation of the 3-injection average for the respective column.</p> <p>Note that the ICV and CCV analyses are also used to monitor RTW drift</p>	<p>If zero, substitute window of close-eluting similar compound. Wider windows can be used to screen for compounds; experience of analyst should weigh heavily in interpretation of chromatograms (refer to RT Shift).</p>
Retention Time (RT) Shift	Each CCV; RT of analytes evaluated against the ICV	Column and compound specific; varies with ICV	<p>Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate.</p> <p>Evaluate data based on a comparison with other standards run during the analytical sequence; consider the RTs for the surrogates and spiked compounds analyzed before and after the sample in question:</p> <ul style="list-style-type: none"> - expand RTW to encompass the shift in compound location - if no peaks are found in the expanded window, report the compound as non-detect - if peaks are present, use the confirmation column to verify identification.

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Analytical Method: SW8082	Parameter: Polychlorinated Biphenyls (PCBs)		Summary of Internal Quality Control (QC) Procedures and Corrective Actions
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per each preparation batch of ≤ 20 samples of like matrix	< RL: MB should not contain any target compounds at or above the reporting limit (RL)	<p>Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action:</p> <ul style="list-style-type: none"> - if a sample contains target compounds at $\geq 10X$ amount found in MB or if target compounds are <u>not</u> detected in the sample, then that sample does not require re-extraction and the results may be reported without qualifications - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at $< 10X$ amount found in MB - if the samples are outside the extraction holding time, then complete an NCR and contact PM for sample disposition. <p>Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.</p>
Blank Spike; BS (Laboratory Control Sample; LCS)	One per batch of 20 samples of like matrix	See Laboratory Limits; recoveries for the spiked compounds must be within the advisory limits	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions or analytical preparation was the cause.

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Analytical Method: SW8082	Parameter: Polychlorinated Biphenyls (PCBs)	Summary of Internal Quality Control (QC) Procedures and Corrective Actions	
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
			<p>If still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCR, and reanalyze all associated samples for the analyte which does not meet criteria.</p> <p>If the samples are outside the extraction holding time, then contact PM via NCR for sample disposition.</p> <p>Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.</p>
Matrix Spike (MS)	One per batch of samples, not to exceed 20 samples of a given matrix.	See Laboratory Limits; recoveries for the spiked compounds should be within advisory limits	<p>Check for documentable errors (e.g., calculations and spike preparation).</p> <p>Check unspiked sample results and surrogate recoveries for indications of matrix effects.</p> <p>If no errors are found, and associated BS (LCS) is within advisory limits, then sample matrix effects are the most likely cause. Note in narrative.</p>
Matrix Spike Duplicate (MSD) or Duplicate	One per batch of samples, not to exceed 20 samples of a given matrix.	<p>See Laboratory Limits: See Matrix Spike for MSD recoveries.</p> <p>RPD's should be within</p>	<p>See Matrix Spike for recoveries.</p> <p>If RPDs for the spiked compounds are not within advisory limits, check for</p>

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Analytical Method: SW8082	Parameter: Polychlorinated Biphenyls (PCBs)	Summary of Internal Quality Control (QC) Procedures and Corrective Actions	
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
		advisory limits	<p>documentable errors (e.g., calculations and spike preparation).</p> <p>Check unspiked sample results and surrogate recoveries for indications of matrix effects.</p> <p>If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/Program/QA Managers.</p>
Surrogate Spike	All field samples, standards and quality control samples	See Laboratory Limits; recoveries should be within advisory limits	<p>Check calculations and spike preparation for documentable errors.</p> <p>If no errors are found, and the surrogate recoveries in the MB and blank spikes are within the advisory limits, then sample matrix effects are the most likely cause.</p> <p>However, any samples with surrogate recoveries significantly below the advisory limits, with no visible chromatographic cause, should be reanalyzed to determine if an injection error was the cause for the low recovery.</p> <p>If the surrogate recoveries in the associated MB and BS are <u>not</u> within advisory limits, and the samples are within the</p>

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Analytical Method: SW8082	Parameter: Polychlorinated Biphenyls (PCBs)		Summary of Internal Quality Control (QC) Procedures and Corrective Actions
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
			<p>holding time, then re-extract and reanalyze all associated samples.</p> <p>If the samples are outside the holding time, then contact the PM via an NCR.</p> <p>Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.</p>
Method Detection Limit (MDL) Study; run at analyte concentrations lower than their reporting limit	As needed; at minimum, annually	Positive result < the analyte reporting limit	<p>Determine the reason for failure and fix problem with system; then repeat study for those analytes that did not meet criteria:</p> <p>- adjust the laboratory reporting limits, if needed.</p>

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STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE COMPOUNDS BY
GAS CHROMATOGRAPHY/MASS SPECTROMETRY
(METHOD 8260B AND METHOD 624)

FORM NUMBERS: NONE

PREPARED BY: Gary Brook		DATE 2/12/99
APPROVED BY: TECHNICAL MANAGER <i>[Signature]</i>		DATE
QUALITY ASSURANCE MANAGER <i>[Signature]</i>		DATE
LABORATORY MANAGER <i>[Signature]</i>		DATE 02/15/99

History: Rev. 0, 3/21/96; Rev. 1, 6/10/96; Rev. 2, 5/May/97; Rev. 3, 13/Apr/98; Rev. 4, 02/15/99.

1. SCOPE AND APPLICATION

- 1.1. This standard operating procedure (SOP) and the method it references, Method 8260B, are used to determine volatile organic compounds in a variety of solid waste matrices. This SOP is applicable to nearly all types of samples, regardless of water content, including: ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

The body of this SOP specifies the procedures to be used for SW-846 Method 8260B. Any additional or contradictory requirements for EPA Method 624 are contained in Section 10.

Parameter	CAS No. ^b	Purge-and-Trap	Direct Injection
Acetone	67-64-1	pp	a
Acrolein	107-02-8	a	a
Acrylonitrile	107-13-1	a	a
Benzene	71-43-2	a	a
Bromobenzene	108-86-1	a	a
Bromochloromethane	74-97-5	a	a
Bromodichloromethane	75-27-4	a	a
Bromoform	75-25-2	a	a
Bromomethane	74-83-9	a	a
2-Butanone (MEK)	78-93-3	pp	a
n-Butyl Benzene	104-51-8	a	a
sec-Butyl Benzene	135-98-8	a	a
tert-Butyl Benzene	98-06-6	a	a
Carbon tetrachloride	56-23-5	a	a
Carbon disulfide	75-15-0	pp	a
Chlorobenzene	108-90-7	a	a

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Parameter	CAS No. ^b	Purge-and-Trap	Direct Injection
Chlorodibromomethane	124-48-1	a	a
Chloroethane	75-00-3	a	a
Chlorohexane		a	a
2-Chloroethyl vinyl ether	110-75-8	a	a
Chloroform	67-66-3	a	a
Chloromethane	74-87-3	a	a
2-Chlorotoluene	95-49-8	a	a
Dibromochloromethane	124-48-1	a	a
4-Chlorotoluene	106-43-4	a	a
1,2-Dibromo-3-chloropropane	96-12-8	pp	
1,2-Dibromoethane	106-93-4	a	
Dibromomethane	74-95-3	a	
1,2-Dichlorobenzene	95-50-1	a	
1,3-Dichlorobenzene	541-73-1	a	
1,4-Dichlorobenzene	106-46-7	a	
Dichlorodifluoromethane	75-71-8	a	
1,1-Dichloroethane	75-34-3	a	
1,2-Dichloroethane	107-06-2	a	
1,1-Dichloroethene	75-35-4	a	
cis-1,2-Dichloroethene	156-59-2	a	a
trans-1,2-Dichloroethene	156-60-5	a	
1,2-Dichloropropane	78-87-5	a	
2,2-Dichloropropane	594-20-7	a	a
1,3-Dichloropropane	142-28-9	a	a
1,1-Dichloropropene	563-58-6	a	a
cis-1,3-Dichloropropene	10061-01-5	a	
trans-1,3-Dichloropropene	10061-02-6	a	
Ethylbenzene	100-41-4	a	
Hexachlorobutadiene	87-68-3	a	
2-Hexanone (MEK)	591-78-6	pp	
Iodomethane	74-88-4	a	
Isopropylbenzene	98-82-8	a	
p-Isopropyltoluene	99-87-6	a	a
Methylene chloride (DCM)	75-09-2	a	
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	

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Parameter	CAS No. ^b	Purge-and-Trap	Direct Injection
Methyl-t-butyl ether (MTBE)	75-97-8	a	a
Naphthalene	91-20-3	a	
n-Propylbenzene	103-65-1	a	a
Styrene	100-42-5	a	a
1,1,1,2-Tetrachloroethane	630-20-6	a	a
1,1,2,2-Tetrachloroethane	79-34-5	a	a
Tetrachloroethene	127-18-4	a	a
Toluene	108-88-3	a	a
1,2,4-Trichlorobenzene	120-82-1	a	a
1,2,3-Trichlorobenzene	87-61-6	a	a
1,1,1-Trichloroethane	71-55-6	a	a
1,1,2-Trichloroethane	79-00-5	a	a
Trichloroethene	79-01-6	a	a
Trichlorofluoromethane	75-69-4	a	a
Trichlorotrifluoromethane	76-13-1	a	a
1,2,3-Trichloropropane	96-18-4	a	a
1,2,4-Trimethylbenzene	95-63-6	a	a
1,3,5-Trimethylbenzene	108-67-8	a	a
Vinyl acetate	108-05-4	a	a
Vinyl chloride	75-01-4	a	a
o-Xylene	95-47-6	a	a
m,p-Xylene	108-38-3	a	a
	106-42-3		

a Adequate response by this technique.

b Chemical Abstract Services Registry Number.

pp Poor purging efficiency resulting in high EQLs.

- 1.2. Method 8260 is based upon a purge-and-trap GC/MS procedure and can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low molecular-

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weight halogenated hydrocarbons, aromatics, ketones, nitrites, acetates, acrylates, ethers, and sulfides

2. SUMMARY

- 2.1. The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection (in limited applications). Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analytes are desorbed directly onto a narrow bore capillary column for analysis. The column is temperature programmed to separate the analytes which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph. Narrow bore capillary columns can be directly interfaced to the ion source.
- 2.2. If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in solvent to dissolve the volatile organic constituents. A portion of the solution is combined with organic-free reagent water in the purge chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method.
- 2.3. Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard with a five-point calibration curve.

3. RESPONSIBILITIES

- 3.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.
- 3.2. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this method. This demonstration may come in the form of

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supervisory/training review, precision and accuracy tests, or the successful completion of an unknown proficiency evaluation test.

- 3.3. Final review and sign off of the data are performed by the department supervisor or designee. Initialing and dating the file indicate that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.
- 3.4. It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

- 4.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter.
- 4.2. Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive technique is rinsing of the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more calibration blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined,

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it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning.

- 4.2.1. The low purging efficiency of many analytes from a 25 mL sample often results in significant concentrations remaining in the sample purge vessel after analysis. After removal of the analyzed sample aliquot and three rinses of the purge vessel with analyte free water, it is required that the empty vessel be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel to reduce sample to sample carryover.
- 4.3. Special precautions must be taken to analyze methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, or random background levels will result. Because methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean because clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.
- 4.4. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol serves as a check on such contamination.
- 4.5. Direct injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. Use of direct injection will result in the need for more frequent instrument maintenance.

5. APPARATUS AND MATERIALS

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5.1. List of Instrumentation

- 5.1.1. Purge and trap device, OI 4560A Liquid Sample Concentrator.
- 5.1.2. Autosampler, OI MPM and Tekmar LSC 2000 and Tekmar ALS 2016 16 port.
- 5.1.3. Gas chromatograph, HP 5890A.
- 5.1.4. Capillary column, Restek RTX-624, 60 m, 0.25 mm ID, 1.4 μ m film thickness (or equivalent)
- 5.1.5. Mass spectrometer, HP5971 MSD or HP5972 MSD.
- 5.1.6. Mass spectral library, National Bureau of Standards (NBS); 98,000 compounds.

5.2. Gas chromatography/mass spectrometer/data system.

5.2.1. Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection or interface to purge-and-trap apparatus. The system includes all required accessories, including syringes, analytical columns, and gases. The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

5.2.2. Gas chromatographic columns

5.2.2.1. Column 1 - 60 m x 0.25 mm ID capillary column coated with DB-VRX (J&W Scientific), 1.5 μ m film thickness, or equivalent.

5.2.2.2. Column 2 - 60 m x 0.25 mm ID capillary column coated with RTX-624 (RESTEK), 1.5 μ m film thickness, or equivalent.

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- 5.2.3. Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for p-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) is injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.
- 5.2.4. GC/MS interface to the mass spectrometer.
- 5.2.4.1. Direct coupling by inserting the column into the mass spectrometer is generally used for 0.25-0.32 mm id columns.
- 5.2.5. Any enrichment device or transfer line can be used if all of the performance specifications described in this SOP (including acceptable calibration at 50 ng or less) can be achieved. GC-to-MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.
- 5.2.6. Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.
- 5.3. Microsyringes - 10, 25, 100, 250, 500, and 1,000 μ L.
- 5.4. Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

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- 5.5. Syringes - 5, 10, or 25 mL, gas-tight with shutoff valve.
- 5.6. Balance - Analytical, 0.0001 g, and top-loading, 0.1 g.
- 5.7. Glass scintillation vials - 20 mL, with Teflon lined screw-caps or glass culture tubes with Teflon lined screw-caps.
- 5.8. Vials - 2 mL.
- 5.9. Disposable pipets - Pasteur.
- 5.10. Volumetric flasks, Class A - 5 mL, 10 mL, and 100 mL, with ground-glass stoppers.
- 5.11. Spatula - Stainless steel.

6. REAGENTS

- 6.1. Reagent grade chemicals shall be used in all tests.
- 6.2. Organic-free reagent water - All references to laboratory-supplied water in this method refers to organic-free reagent water.
- 6.3. Methanol, CH₃OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.
- 6.4. Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.
- 6.5. Stock solutions - NIST traceable stock solutions are purchased from multiple vendors as certified solutions. Concentrations of stock solutions vary from 1,000-10,000 ug/mL.

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6.5.1. Transfer the stock standard solution into a bottle with a Teflon-lined screw-cap. Store, with minimal headspace, at -10°C to -20°C and protect from light.

6.5.2. *Gas stock solutions expire one month* after the ampule has been opened and transferred to a Teflon-lined screw cap vial. *Other calibration stock solutions expire three months* after the ampule has been opened and transferred to a Teflon-lined screw cap vial.

6.5.2.1.1. Optionally, calibration using a certified gaseous mixture can be accomplished daily utilizing commercially available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichlorodifluoromethane and trichlorofluoromethane in nitrogen. These mixtures of documented quality are stable for as long as six months without refrigeration.

6.5.3. DOCUMENTATION

All standards preparation information is to be fully documented in a standards prep logbook. Information, such as manufacturer, compound, analyst, date prepared, solvent used, aliquot volume, date received, date opened, and final concentration is to be recorded.

6.6. Secondary dilution (working level) standards - Using stock standard solutions, prepare in methanol, secondary dilution standards containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. *Store in a vial with no headspace for one week only.*

6.7. Surrogate standards - The surrogates used for this method are: toluene-d8, 4-bromofluorobenzene, 1,2-dichloroethane-d4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements.

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A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250 µg/mL in methanol. Each water sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis.

- 6.8. Internal standards - The internal standards used for this method are: pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described above. It is recommended that the secondary dilution standard should be prepared at a concentration of 50 mg/L of each internal standard compound. Addition of 5 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L.
- 6.9. 4-Bromofluorobenzene (BFB) standard - A standard solution containing 50 ng/µL of BFB in methanol is be prepared.
- 6.10. Calibration standards - Calibration standards at a minimum of five concentrations should be prepared from the secondary dilution of stock standards. Prepare these solutions in organic-free reagent water. One of the concentrations should be at a concentration less than or equal to the reporting limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples but should not exceed the working range of the GC/MS system. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s). ***Calibration standards must be prepared daily.***
- 6.11. Matrix spiking standards - Matrix spiking standards should be prepared from volatile organic compounds which will be representative of the compounds being investigated. At a minimum, the matrix spike will include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The standard is prepared in methanol, with each compound present at a concentration of 25 ug/mL.

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- 6.12. Great care must be taken to maintain the integrity of all standard solutions. It is recommended that all standards in methanol be stored at -10°C to -20°C in amber bottles with Teflon lined screw-caps.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

- 7.1. Samples must be collected according to an approved sampling plan.
- 7.2. Volatile organic analysis of water and soil samples must be performed within 14 days of collection unless otherwise specified by the client. Water samples are usually preserved by adding approximately four (4) drops of concentrated hydrochloric acid to each 40 mL vial. The purpose of the hydrochloric acid is to prevent microbial degradation of aromatic compounds. If the water sample is unpreserved, the holding time may be shortened to seven (7) days from the date of collection.
- 7.3. Measure and record the pH of each aqueous sample immediately before analysis. Notify the Project Manager immediately if the pH of the sample is greater than 2.
- 7.4. Samples must be collected in glass containers without headspace and stored at $4 \pm 2^\circ \text{C}$.
- 7.5. To prevent loss of volatile organic compounds, samples must not be opened until the time of analysis.

8. PROCEDURE

- 8.1. Three alternate methods are provided for sample introduction. All internal standards, surrogates, and matrix spikes (when applicable) must be added to samples before introduction.
- 8.1.1. Direct injection - in very limited application, (e.g., volatiles in waste oil or aqueous process wastes) direct injection of aqueous samples or samples diluted according to Method 3585 may be appropriate. Direct injection has been used for the analysis of volatiles in waste oil (diluted 1:1 with hexadecane) and for determining if the sample is ignitable (aqueous

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injection, Methods 1010 or 1020). Direct injection is only permitted for the determination of volatiles at the toxicity characteristic (TC) regulatory limits, at concentrations in excess of 10,000 µg/L, or for water-soluble compounds that do not purge.

8.1.2. Purge-and-trap for aqueous samples.

8.1.3. Purge-and-trap for solid samples.

8.2. Recommended instrument conditions (typical).

8.2.1. Purge and trap settings for OI 4560A Purge and Trap Device:

sparge time = 6-11 minutes

desorb temperature = 240 °C.

desorb time = at least 1.5 minute.

trap bake = at least 8 minutes at 260 °C.

8.2.2. Purge and trap settings for Tekmar LSC 2000:

sparge time = 6-11 minutes

desorb temperature = 250 °C.

desorb time = at least 6 minutes.

trap bake = at least 4 minutes at 260 °C.

8.2.3. GC/MS operating conditions:

initial temperature = 60 °C.

initial time = 0.1 minute.

temperature ramp A = 10 °C/minute .

temperature ramp B = 25 °C/minute.

final temperature A = 105 °C.

final temperature B = 220 °C.

final hold time A = 0 minutes.

final hold time B = until all compounds elute.

transfer line temperature = 120 °C.

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injection temperature = 150 °C

energy = 70 eV (nominal).

mass range = 35 - 260 amu.

scan time = 6 scans per peak, not to exceed 1 second per scan.

- 8.2.4. Each GC/MS system must be hardware-tuned to meet the criteria in Table 1 for a 5-50 ng injection or purging of 4-bromofluorobenzene (1 µL injection of the BFB standard). A BFB tune is performed prior to analysis to demonstrate the ability of the system to separate ions and assign proper ratios to fragments. Analyses must not begin until these criteria are met. One microliter (1 µL) of a 50 ng/µL solution of BFB is analyzed by direct injection.
- 8.2.5. Set up the purge-and-trap system as outlined in Method 5030 if purge-and-trap analysis is to be utilized. A set of at least five calibration standards containing the method analytes and surrogates is needed. One calibration standard should contain each analyte at a concentration approaching but greater than the method detection limit for that compound. The other calibration standards should contain analytes at concentrations that define the range of the method.
- 8.2.6. Calibration should be done using the sample introduction technique that will be used for samples. The purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.
- 8.2.6.1. To prepare a calibration standard for purge-and-trap or aqueous direct injection, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight

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syringe along with 10 µL of internal standard. Then transfer the contents to a purging device or syringe. Perform purge-and-trap or direct injection as outlined in Method 5030.

8.2.6.2. To prepare a calibration standard for direct injection analysis of oil, dilute standards in methanol.

8.2.7. Tabulate the area response of the characteristic ions (see Table 2) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = (A_x C_{IS}) / (A_{IS} C_x)$$

where:

A_x = Area of the characteristic ion for the compound being measured.

A_{IS} = Area of the characteristic ion for the specific internal standard.

C_{IS} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

8.2.7.1. The average RF must be calculated and recorded for each compound using at least five RF values calculated for each compound from the initial calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average relative response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane; and chlorobenzene. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

8.2.7.2. Chloromethane - This compound is the most likely compound to be lost if the purge flow is too fast.

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8.2.7.3. Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by tuning of BFB at ions m/z 174/176. Increasing the m/z 175/176 ratio relative to m/z 95 may improve bromoform response.

8.2.7.4. Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

8.2.8. Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all compounds. The percent RSD is calculated as follows:

$$\%RSD = \frac{SD}{RF_x} \times 100\%$$

where:

RSD = Relative standard deviation
RF_x = mean of 5 initial RFs for a compound
SD = Standard deviation of the 5 initial RFs for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}}$$

where:

Rf_i = RF for each of the 5 calibration levels
N = number of RF values (i.e., 5)

The percent relative standard deviation should be less than 15% for each compound. However, the %RSD for each individual

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Calibration Check Compound (CCC) must be less than 30%. The CCCs are:

1,1-Dichloroethene,
Chloroform,
1,2-Dichloropropane,
Toluene,
Ethylbenzene, and
Vinyl chloride.

- 8.2.9. If the %RSD of any compound is greater than 30 percent is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is required before attempting another calibration.
- 8.2.10. Linearity - If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.
- 8.2.11. If the %RSD of any compound is greater than 15%, construct calibration curves of area ratio (A/AIS) versus concentration using first or higher order regression fit of the five calibration points. The analyst should select the regression order which introduces the least calibration error into the quantitation. The use of calibration curves is a recommended alternative to average response factor calibration and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.
- 8.2.12. In those instances where the RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the mean of the RSD values for all analytes in the calibration is $\leq 15\%$.
- 8.2.13. These curves are verified each shift by purging a continuing calibration standard. Recalibration is required only if calibration and on-going performance criteria cannot be met.

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- 8.3. GC/MS calibration verification (3 steps, performed at the beginning of each 12 hour sequence)
- 8.3.1. Prior to the analysis of samples, inject or purge 5-50 ng of the 4-bromofluorobenzene standard following Method 5030. The resultant mass spectra for the BFB must meet all of the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift.
- 8.3.2. The initial calibration curve for each compound of interest must be checked and verified once every 12 hours during analysis with the introduction technique used for samples. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC and CCC.
- 8.3.3. System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum relative response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.
- 8.3.3.1. The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.1
1,1-Dichloroethane	0.1
Bromoform	0.10
Chlorobenzene	0.3
1,1,2,2-Tetrachloroethane	0.3

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- 8.3.4. Calibration Check Compounds (CCCs) - After the system performance check is met, the CCCs listed are used to check the validity of the initial calibration. Calculate the percent difference using the following equation:

$$\% \text{ Difference} = (RF - RF_1)/RF \times 100$$

where:

RF₁ = Calibration Check Compound standard response factor.
RF = Average response factor.

If the percent difference for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% difference), for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If the source of the problem can not be determined after corrective action has been taken, a new five point calibration must be generated. This criterion **MUST** be met before quantitative sample analysis begins. If the CCCs are not required analytes by the permit, then all required analytes must meet the 20% drift criterion.

- 8.3.5. The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections, must be made as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration check standard, the mass spectrometer must inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

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8.4. GC/MS analysis.

8.4.1. All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the purge-and-trap system as outlined in Method 5030 if purge-and-trap introduction will be used.

8.4.2. BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

8.4.2.1. Remove the plunger from a 5 mL syringe and attach a closed syringe valve. If lower detection limits are required, use a 25 mL syringe. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL.

8.4.3. The process of taking an aliquot destroys the validity of aqueous and soil samples for future analysis; therefore, if there is only one VOA vial, the analyst should prepare a second aliquot for analysis at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, filling one 20 mL syringe would require the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

8.4.3.1. The following procedure is appropriate for diluting aqueous purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

8.4.3.1.1. Dilutions may be made in volumetric flasks (10 to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

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8.4.3.1.2. Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

8.4.3.1.3. Inject the proper aliquot of sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

8.4.3.1.4. Fill a 5 mL syringe with the diluted sample.

8.4.3.2. Compositing aqueous samples prior to GC/MS analysis

8.4.3.2.1. Add 5 mL or equal larger amounts of each sample (up to 5 samples are allowed) to a 25 mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe.

8.4.3.2.2. The samples must be cooled at 4 °C during this step to minimize volatilization losses.

8.4.3.2.3. Mix well and draw out a 5 mL aliquot for analysis.

8.4.3.2.4. Follow sample introduction, purging, and desorption steps described in Method 5030.

8.4.3.2.5. If less than five samples are used for compositing, a proportionately smaller syringe may be used unless a 25 mL sample is to be purged.

8.4.4. Add 5 µL of surrogate and internal standard to each sample. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 5 µL of the surrogate spiking solution to 5 mL of sample is equivalent to a concentration of 50 µg/L of each surrogate

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standard. The addition of 5 μL of the surrogate spiking solution to 5 g of sample is equivalent to a concentration of 50 $\mu\text{g}/\text{kg}$ of each surrogate standard.

- 8.4.5. Perform purge-and-trap or direct injection by Method 5030. If the initial analysis of sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank organic-free reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

8.4.5.1. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

- 8.4.6. For matrix spike analysis, add 10 μL of the matrix spike solution to the 5 mL of sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 50 $\mu\text{g}/\text{L}$ of each matrix spike standard.

- 8.4.7. Heated Purge.

8.4.7.1. The initial calibration curve, all continuing calibration standards, and all field and quality control samples shall be heated to 40 $^{\circ}\text{C}$ during the purge.

- 8.4.8. Unheated Purge.

8.4.8.1. The initial calibration curve, all continuing calibration standards, and all field and quality control samples shall not be heated during the purge.

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8.4.9. Water Samples.

8.4.9.1. Five (5) or twenty five (25) milliliters of sample are placed in a gas-tight syringe and spiked with 10 uL of 25 ng/uL internal standard and surrogate solution. The sample is placed in a sparge tube on the autosampler. In an attempt to improve laboratory efficiency, water samples can be heated along with soil samples.

8.4.9.2. Sample dilution is based on analyte concentration, unknown compound concentration, or the presence of surfactants (foaming samples).

8.4.9.3. If surrogate recoveries fall outside control limits, then the sample must be reanalyzed unless there is an obvious interference such as a large amount of coeluting material.

8.4.10. Soil Samples.

8.4.10.1. Five grams of soil is weighed into a 5 mL sparge tube. The sample is placed on the autosampler. Then add 5 mL of the reagent water and 5 uL of internal standard and surrogate solution to the soil. No less than 0.5 g of soil should be purged.

8.4.10.2. Sample dilution is based on analyte concentration or unknown compound concentration. Whatever dilution is made, the results will be multiplied by this dilution factor.

8.4.10.3. Medium Level Soil Extraction.

8.4.10.3.1. Soil samples requiring less than 0.5 g to be purged must be analyzed using a medium level extraction technique.

8.4.10.3.2. Mix entire contents of sample. For soil or sediments insoluble in methanol, weigh 4 g of sample into a tared 20 mL vial. Add 10 mL of methanol and shake for one minute. After contents have settled, remove 100 uL of the

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methanol extract and deliver into 4.9 mL of reagent water.
Add internal standard and surrogate solution and analyze.

8.4.10.3.3. A medium level blank consisting of 100 uL of methanol is to be analyzed before the sample extract, to ensure no methanol contamination.

8.4.10.3.4. This 100 uL methanol extract delivered into 4.9 mL of reagent water results in a 1:50 dilution for the sample. Analytical results and reporting limits are then raised by a factor of 50x.

8.5. Data interpretation.

8.5.1. Qualitative analysis.

8.5.1.1. The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

8.5.1.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

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8.5.1.1.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

8.5.1.1.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

8.5.1.1.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

8.5.1.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulders or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

8.5.1.1.6. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

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8.5.1.2. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

- (1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within $\pm 20\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library

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searches will the mass spectral interpretation
specialist assign a tentative identification. .

8.5.2. Quantitative analysis.

8.5.2.1. When a compound has been identified the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte.

8.5.2.2. When MS response is linear and passes through the origin, calculate the concentration of each identified analyte in the sample as follows:

Water;

$$\text{Concentration}(\mu\text{g} / \text{L}) = \frac{(A_x)(I_s)}{(A_{IS})(RF)(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured.

I_s = Amount of internal standard injected (ng).

A_{IS} = Area of characteristic ion for the internal standard.

RF = Mean relative response factor for compound being measured.

V_o = Volume of water purged (mL), taking into consideration any dilutions made.

Sediment/Soil Sludge (on a dry-weight basis) and Waste
(normally on a wet-weight basis);

$$\text{Concentration}(\mu\text{g} / \text{kg}) = \frac{(A_x)(I_s)(V_i)}{(A_{IS})(RF)(V_i)(W_s)(D)}$$

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where:

$A_x, I_s, A_{is}, \overline{RF}$ = Same as for water.

V_t = Volume of total extract (μL)
(Use 10,000 μL or a factor of this when dilutions are made).

V_i = Volume of extract added (μL) for purging.

W_s = Weight of sample extracted or purged (g).

D = % dry weight of sample/100, or 1 for a wet-weight basis.

8.5.2.3. Where requested by the client, an estimate of concentration for noncalibrated components in the sample may be made. The formulae given above should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

8.5.2.4. Alternatively, the regression line fitted to the initial calibration may be used for determination of analyte concentration.

9. QUALITY CONTROL

9.1. Instrument criteria

9.1.1. The GC/MS system must be tuned to meet the BFB specifications.

9.1.2. There must be an initial calibration of the GC/MS system.

9.1.3. The GC/MS system must meet the SPCC criteria and the CCC criteria, each 12 hours.

9.2. Surrogate recovery is monitored to assess method performance the particular matrix. Surrogates are added to all samples and blanks prior to analysis.

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Surrogates are added to all initial and continuing calibration standards. For aqueous and soil matrices, laboratory established surrogate control limits should be compared with the control limits listed in Table 3.

9.2.1. If recovery is not within limits, the following procedures are required.

9.2.1.1. Check to be sure that there are no errors in the calculations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.2.1.2. Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract.

9.2.1.3. If no problem is found, re-extract and re-analyze the sample.

9.2.1.4. If, upon re-analysis, the recovery is again not within limits, then narrate the surrogate discrepancy and submit both sets of data.

9.2.2. At a minimum, the laboratory should update surrogate recovery limits on a matrix-by-matrix basis, annually.

9.3. BLANK ANALYSIS

9.3.1. To verify that system interferences are minimized, a reagent blank must be analyzed for each 12-hour BFB tune and per batch of 20 or fewer field samples. Target compounds may not be detected above the reporting limit. Common laboratory contaminants, such as acetone, 2-butanone and methylene chloride, are allowed at levels as high as five times the reporting limit. This laboratory contamination must be reported in the case narrative and should be considered a warning for laboratory contamination.

9.3.2. If the method blanks contains target compounds above the reporting limits, then the analytical system is considered out of control. Sample analysis

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may not continue until a clean method blank has been acquired. Document the situation and its resolution on a corrective action form (NCR).

9.4. MATRIX SPIKE SAMPLES

9.4.1. A matrix spike (MS) and matrix spike duplicate (MSD) sample are analyzed to evaluate the effect of the matrix. The frequency of the MS/MSD shall be one pair per batch of 20 field samples.

10. DEVIATIONS FROM METHOD

10.1. This SOP meets the requirements of Method 8260B. There are no known deviations from the method.

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10.2. EPA METHOD 624

- 10.2.1. The items contained in this Section are differences between Method 8260B and Method 624. The issues in this section supersede any contradictory requirements set forth in the remainder of this SOP.
- 10.2.2. Suggested internal standards and surrogates are listed in Method 624, Table 3. Paragon uses the same internal standards and surrogates for Methods 8260B and 624 (internal standards: pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4; surrogates: toluene-d8, 4-bromofluorobenzene, 1,2-dichloroethane-d4, and dibromofluoromethane).
- 10.2.3. Method 624 requires a purge time of 11.0 ± 0.1 min. at ambient temperature.
- 10.2.4. Method 624 requires a desorb time of 4 min.
- 10.2.5. Initial Calibration - Although Method 624 permits as few as three points in the initial curve, Paragon will quantitate from a 5-7 point curve.
- 10.2.6. Method 624 states that if the linearity is less than 35 %RSD, an average response factor can be used. Otherwise, construct a linear curve with a correlation coefficient less than 0.995.
- 10.2.7. Method 624 specifies that BFB must be analyzed and meet criteria "each working day." Method 8260B specifies BFB be passed every 12 hours.
- 10.2.8. A continuing calibration verification (CCV) must be performed every working day rather than every 12 hours. Also, the results of the CCV must meet the requirements set forth by Table 5 in the EPA Method 624. Any compounds without limits in this table must have their recovery reported, but corrective actions are not required.
- 10.2.9. A matrix spike (MS) sample must be performed on every 20 samples. The native sample only needs to be spiked once; a matrix spike duplicate

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(MSD) sample is not required. Also, all matrix spikes and blank spikes must contain every analyte of interest.

10.2.10. A set of 4 QC Check samples must be analyzed by an analyst before any samples are processed to demonstrate the ability to perform the method. The concentrations of each compound must be 20 ug/L. The results must fall within the acceptance criteria specified in Table 5 of EPA Method 624.

10.2.11. The matrix spikes and blank spikes must meet the acceptance criteria listed in the Table 5 copied directly from the Method. Note that not all compounds have acceptance limits in this table. For these compounds, the recovery must be reported; however, corrective actions based on those results are not required.

10.3. DEVIATIONS FROM EPA METHOD 624

10.3.1. Because samples from several sites are usually batched together, only one spiking level is used for each compound. It is impractical to match each compound's spike amount with the amount of the compound in the samples chosen for spiking and also matching the spike amount to the appropriate regulatory level for each compound. This difference must be stated in the Case Narrative that accompanies each batch of samples.

11. SAFETY HAZARDS AND WASTE

11.1. LABORATORY SAFETY AND HAZARDS

11.1.1. Environmental samples may contain unknown hazards. Personal protective equipment must be worn at all times. Personal protective equipment shall consist of safety glasses, labcoat, and gloves. Please consult the MSDS or the Health and Safety Coordinator for more information.

11.2. WASTE DISPOSAL

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11.2.1. The aqueous phase of the purge and trap waste shall be disposed on in the aqueous lab waste stream. A satellite waste collection vessel may be obtained from the Waste Disposal Coordinator. The solid phase of the purge and trap waste shall be disposed of in the contaminates soils and solids waste stream. A satellite waste collection vessel(s) may be obtained from the Waste Disposal Coordinator.

12. REFERENCES

- 12.1. Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water Method 524.2; US Environmental Protection Agency. Office of Research Development, Environmental Monitoring and Support Laboratory, Cincinnati, OH 1986.,
- 12.2. Method 624 Purgeables, Federal Register, Volume 49, Number 209, October 26, 1984.
- 12.3. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Method 8260B, Volatile organic Compounds by Gas Chromatography/Mass Spectrometry, Revision 2, December 1996.

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TABLE 1

BFB MASS INTENSITY SPECIFICATIONS (4-BROMOFLUOROBENZENE)*

<u>MASS</u>	<u>INTENSITY REQUIRED (relative abundance)</u>
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

* Alternate tuning criteria may be used (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

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TABLE 2

CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS

<u>TARGET ANALYTE</u>	<u>PRIMARY CHARACTERISTIC ION(S)</u>	<u>SECONDARY CHARACTERISTIC ION(S)</u>
Acetone	43	58
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Benzene	78	52, 77
Bromobenzene	77	156, 158
Bromochloromethane	49	128, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
1-Chlorohexane	91	55, 93
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	127	129
1,2-Dibromomethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148

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<u>TARGET ANALYTE</u>	<u>PRIMARY CHARACTERISTIC ION(S)</u>	<u>SECONDARY CHARACTERISTIC ION(S)</u>
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	61	53, 96
cis-1,2-Dichloroethene	61	96, 98
trans-1,2-Dichloroethene	61	96, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
Ethylbenzene	91	106
Hexachlobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Iodomethane	142	127, 141
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methyl-t-butyl ether	73	57
Methylene chloride	49	86, 84
4-Methyl-2-pentanone	43	58, 85, 100
Naphthalene	128	
n-Propylbenzene	91	120
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachlorobenzene	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	129, 131, 164

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<u>TARGET ANALYTE</u>	PRIMARY CHARACTERISTIC ION(S)	SECONDARY CHARACTERISTIC ION(S)
Toluene	91	92
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	97	83, 85
Trichloroethane	95	97, 130, 132
Trichlorofluoromethane	101	151, 153
1,2,3-Trichloropropane	75	77
Trichlorotrifluoroethane	101	103, 151, 153
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	91	106
m-Xylene	91	106
p-Xylene	91	106

INTERNAL STANDARDS (IS) /SURROGATES (SS)

1,4-Difluorobenzene IS	114	
Chlorobenzene-d ₅ IS	117	
1,4-Dichlorobenzene-d ₄ IS	152	115, 150
Pentafluorobenzene IS	168	
Dibromofluorobenzene SS	113	
Toluene-d ₈ SS	98	
1,2-dichloroethane-d ₄ SS	65	
4-Bromofluorobenzene SS	95	174, 176

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TABLE 3

**SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT
SAMPLES -- LIMITS FROM METHOD 8260B**

<u>SURROGATE COMPOUND</u>	<u>Appropriate Technique</u>	
	<u>Low/High Water</u>	<u>Low/High Soil/Sediment</u>
4-Bromofluorobenzene ^a	86-115	74-121
Dibromofluoromethane ^a	86-118	80-120
Toluene-d ₈	88-110	81-117
1,2-Dichlorooctane-d ₄	80-120	80-120

^a Single laboratory data, for guidance only

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Analytical Method: SW8260B	Parameter: Volatile Organic Compounds	TABLE 4. Volatiles Summary of Internal Quality Control (QC) Procedures and Corrective Action	
		QC Check	Frequency
		Acceptance Criteria	Corrective Action
Tuning Criteria	Every 12 hour period	BFB breakdown	• Retune. <u>Do not</u> proceed with analysis until tune meets criteria.
Initial Calibration	When CCCs and SPCCs in the daily calibration do not meet criteria (mid-point required for quantitation of all samples analyzed during the 12 hour sequence)	CCC: $\pm 30\%$ RSD; non-CCC: $\pm 15\%$ RSD; SPCC: <i>Chloromethane</i> ≥ 0.10 RF, <i>1,1DCA</i> ≥ 0.10 <i>Bromoform</i> ≥ 0.10 <i>Chlorobenzene</i> ≥ 0.30 <i>1,1,2,2-tetrachloroethane</i> ≥ 0.30	• for CCC and SPCC, reanalyze the initial calibration curve and/or evaluate/correct instrument malfunction to obtain curve which meets criteria.
Daily Calibration (mid-point)	Every 12 hour period following tune (required for quantitating all samples analyzed during the 12 hour sequence)	CCC: $\pm 20\%$ D;	• Reanalyze the daily standard. If still out, evaluate/correct instrument malfunction as needed; initiate a new calibration curve.
Method Blank	Every 12 hour period After each calibration	< RL for all target compounds, except common laboratory contaminants (methylene chloride, acetone, 2-butanone)	• reanalyze to determine if instrument contamination was the cause. If the method blank is still non-compliant, correct the problem before analysis of samples.
Matrix Spike (MS)	1 per batch of samples, not to exceed 20 samples of a given	See Laboratory Limits: The recoveries for the spiked compounds should	• if non-compliant, check calculations and spike preparation for documentable

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Analytical Method: SW8260B		Parameter: Volatile Organic Compounds		TABLE 4. Volatiles Summary of Internal Quality Control (QC) Procedures and Corrective Action	
QC Check		Frequency	Acceptance Criteria	Corrective Action	
		matrix.	be within advisory limits.	errors. • if no errors are found, and the associated blank spike is within control limits, then sample matrix effects are the most likely cause.	
Matrix Spike Duplicate (MSD) or Duplicate		1 per batch of samples, not to exceed 20 samples of a given matrix.	See Laboratory Limits: See Matrix Spike for recoveries. RPD's should be within advisory limits.	• if non-compliant, check calculations for documentable errors. • check unspiked sample results and surrogate recoveries for indications of matrix effects. • if significant differences (>15%) exist between the MS and MSD (or between duplicates) reanalysis of the sample and spikes may be necessary.	

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**Analytical
Method:**
SW8260B

Parameter:
Volatile Organic Compounds

TABLE 4. Volatiles
**Summary of Internal Quality
Control (QC) Procedures and
Corrective Action**

QC Check	Frequency	Acceptance Criteria	Corrective Action
Blank Spikes (BS)	1 for each MS/MSD outside control limits	See Laboratory Limits: The recoveries for the spiked compounds should be within advisory limits.	<ul style="list-style-type: none"> • if non-compliant, check calculations and spike preparation for documentable errors. • if no errors are found, then reanalyze the blank spike to determine if instrumental conditions or analytical preparation was the cause. Notify the Supervisor and initiate corrective action (NCR). • reanalyze associated samples, if appropriate.
Surrogate Spikes	Every sample	See Laboratory Limits: The recoveries for the spiked compounds should be within advisory limits.	<ul style="list-style-type: none"> • if non-compliant, check calculations and spike preparation for documentable errors. • reanalyze sample once (re-analysis requirements may be fulfilled by existing multiple analyses, e.g., MS, MSD, REP, sample dilutions). If still out, report results and note in narrative.

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Analytical Method: SW8260B	Parameter: Volatile Organic Compounds	TABLE 4. Volatiles Summary of Internal Quality Control (QC) Procedures and Corrective Action	
QC Check	Frequency	Acceptance Criteria	Corrective Action
Internal Standard (IS)	Every sample, standard and blank	Average area within -50% to +100% window	<ul style="list-style-type: none"> • inspect instrument for malfunction; correct identified malfunctions, then reanalyze samples. • if no instrument malfunction identified and CLP QC, reanalyze <ul style="list-style-type: none"> - if out-of-limit areas are explained by the sample matrix, reanalysis will not be required (e.g., high hydrocarbon content contributes to IS areas) - re-analysis requirements may be fulfilled by existing multiple analyses (e.g., MS, MSD, REP, sample dilutions)
Retention Time Shift (RT)	Every sample, standard, and blank	RT shift <30 seconds compared to daily standard (STD50) Relative retention time (RRT) of sample must be ± 0.06 RRT units of standard	<ul style="list-style-type: none"> • inspect chromatographic system for malfunction; correct identified malfunctions, then reanalyze sample.
Precision and Accuracy Study: (minimum of 4 replicate analyses of a QC check	One-time demonstration per method	method specified limits, if available (refer to QC Acceptance Criteria Table in the respective method)	<ul style="list-style-type: none"> • check calculated results for error • determine the reason for failure and fix problem with

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**Analytical
Method:**
SW8260B

Parameter:
Volatile Organic Compounds

**TABLE 4. Volatiles
Summary of Internal Quality
Control (QC) Procedures and
Corrective Action**

QC Check	Frequency	Acceptance Criteria	Corrective Action
sample)			system; then repeat study for those analytes that did not meet criteria
Method Detection Limit (MDL) Study	Annually	Value must be < reporting limit	<ul style="list-style-type: none">• determine the reason for failure and fix problem with system; then repeat study for those analytes that did not meet criteria• adjust the laboratory reporting limits, if needed

ENCLOSURE 7:

Paragon Control Charts

TARGET SHEET
EPA REGION VIII
SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 494266

SITE NAME: VASQUEZ BOULEVARD/INTERSTATE 70

DOCUMENT DATE: 08/16/2001

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☒ TYPES OF DOCUMENTS NOT TO BE SCANNED
(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

CONTROL CHARTS BY ANALYTICAL TEST
